Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Synthesis and Conformational Structure of Hydrazo-Bridged Homo Calix[2]pyridine[2]triazines

Dong-Dong Liang, Mei-Xiang Wang*

†The Key Laboratory of Bioorganic Phosphorous and Chemical Biology (Ministry of Education), Department of Chemistry, Tsinghua University, Beijing 100084 China

E-mail: wangmx@mail.tsinghua.edu.cn

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1. General Information

Unless otherwise noted, all reactions were carried out in oven-dried glasswares. Anhydrous solvents were purified and dried following standard procedures. All commercially available reagents were used as received. Compounds **1**[1], **2a**[2], **8**[3] were synthesized according to published procedures. TLC analysis was performed on pre-coated, glass-backed silica gel plates and visualized with UV light. Unless otherwise noted, flash column chromatography was performed on silica gel (200 - 300 mesh).

Melting points were uncorrected. The ^{1}H NMR, ^{13}C NMR spectra were recorded on a 400 MHz NMR spectrometer. ^{1}H and ^{13}C NMR chemical shifts were reported relative to the signals of residual of chloroform (7.26 ppm) DMSO (2.50ppm) and that of internal standard of TMS (0.0 ppm), respectively. Abbreviations are used in the description of NMR data as follows: chemical shift (δ, ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, dt = doublet of triplets, m = multiplet), coupling constant (*J*, Hz). The electron spray ionization mass spectra (ESI-MS) were recorded on a MASS spectrometer. Infrared spectra were recorded with KBr pellets in the 4000-400 cm^{-1} region.

2. Synthesis

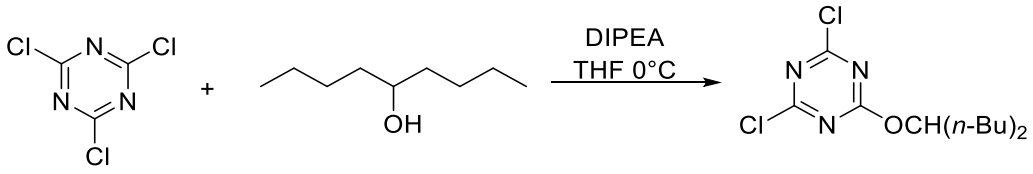
2.1 Synthesis of 1.

2,6-Bis-hydrazinopyridine **1** was synthesized according to literature.[1] Hydrazine hydrate (12.5 mL, 200 mmol, 20 equiv.) was added to a 25 mL oven-dried, argon-filled pear flask equipped with a magnetic stirring bar. With rapid stirring, 2,6-difluoropyridine (1.15g, 10 mmol, 1 equiv.) was added very slowly and cautiously. The reaction mixture was then heated to 80 ºC for 24 h. Upon cooling, a yellow solid formed. Filtration of the resulting suspension, washing with isopropanol and diethyl ether, to give **1** (1 g, 72% yield).

General procedure for the synthesis 2b-c:

To an ice-bath cooled solution of cyanuric chloride **6** (4.05 g, 20 mmol) in THF (50 mL) was added, respectively, alcohol (22 mmol) and 2.9 mL of collidine. The reaction mixture was stirred for another 12 hours and then hydrochloric acid salt of collidine formed during the reaction was filtered off and filtrate was poured into ice-water mixture and extracted with CH_2Cl_2 (3 x 50 mL). After drying (Na_2SO_4), the solvent was removed and the residue was purified by column chromatography on a silica gel column with a mixture of ethyl acetate and petroleum ether as the mobile phase to give pure **2b-c**.

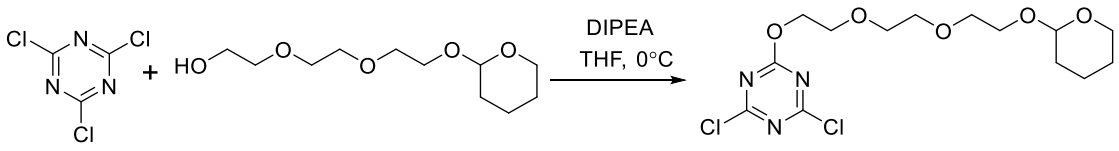
2.1 Synthesis of 2b.



6 **7** **2b**

2b was obtained from flash column chromatographed on a silica gel column (petroleum ether and ethyl acetate = 50 / 1) (2.63g, 45% yield): colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.29-5.26 (m, 1H), 1.78-1.63 (m, 4H), 1.39-1.25 (m, 8H), 0.89 (t, J = 6.9Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.4, 171.1, 81.9, 33.2, 27.2, 22.5, 13.9; IR (KBr, cm^{-1}) ν 2958, 2933, 2863, 1541, 1503. HRMS (APCI-ion trap) calcd. for $C_{12}H_{20}Cl_2N_3O$: $[M+H]^+$ 292.0978. Found: 292.0975.

2.2 Synthesis of 2c.



6 **8** **2c**

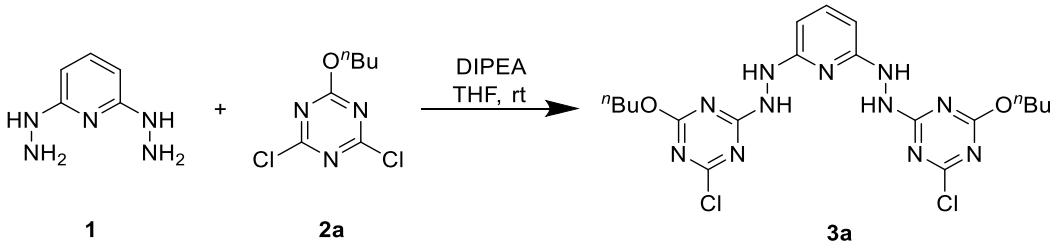
2c was obtained from flash column chromatographed on a silica gel column (petroleum ether and ethyl acetate = 5 / 1) (5.73 g, 75% yield): colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.57-4.49 (m, 3H), 13.78-3.35 (m, 12H), 1.74-1.38 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.3, 171.0, 98.7, 70.7, 70.5, 70.4, 69.3, 68.3, 66.5, 62.0,

30.4, 25.3, 19.3; IR (KBr, cm^{-1}) ν 2941, 2870, 1545, 1511. HRMS (ESI-ion trap) calcd. for $C_{14}H_{21}Cl_2N_3O_5Na$: $[M+Na]^+$ 404.0750. Found: 404.0747.

General procedure for the synthesis 3a-c:

To a solution of 2,6-dihydrazinylpyridine **1** (2 mmol) in tetrahydrofuran (30 mL) was added dropwise a mixture of substituted 2,4-dichloro-1,3,5-triazine **2a-c** (4 mmol) and diisopropylethylamine (4.4 mmol) in tetrahydrofuran (20 mL) during 1 h. The reaction mixture was stirred overnight. After removal of diisopropylethylamine hydrochloride salt through filtration, the filtrate was extracted with ethyl acetate (3 x 50 mL). After drying (Na_2SO_4) and then removing the solvent, the residue was chromatographed on a silica gel column with a mixture of petroleum ether and ethyl acetate as the mobile phase to give pure linear trimer **3a-c**.

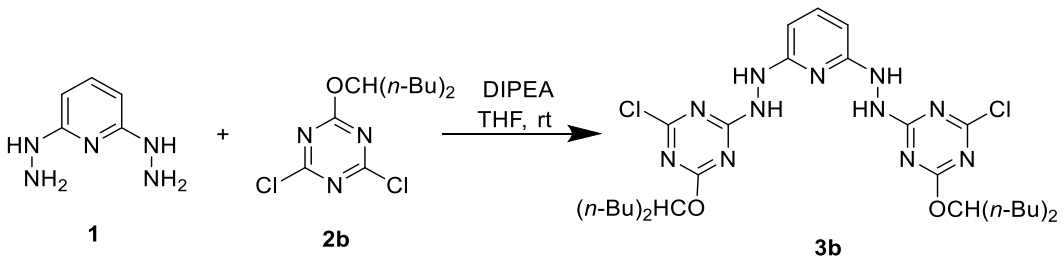
2.3 Synthesis of 3a.



1 2a 3a

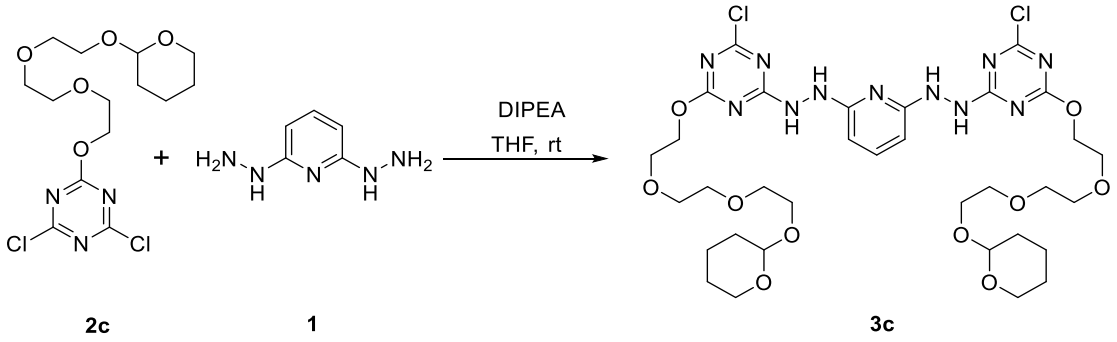
3a was obtained from flash column chromatographed on a silica gel column (petroleum ether and ethyl acetate = 8 / 1) (753.6 mg, 74% yield): white solid, mp 105-107 °C; ^{1}H NMR (400 MHz, $C_2D_2Cl_4$, 120°C) δ 7.76 (br, 2H), 7.41 (t, J = 7.8Hz, 1H), 6.86 (br, 2H), 6.23 (d, J = 7.8Hz, 2H), 4.34 (t, J = 6.9Hz, 4H), 1.74-1.68 (m, 4H), 1.46-1.37 (m, 4H), 0.95 (t, J = 7.3Hz, 6H); ^{13}C NMR (100 MHz, $C_2D_2Cl_4$, 120°C) δ 171.6, 171.3, 169.1, 157.8, 140.5, 99.2, 68.9, 30.7, 19.0, 13.6; IR (KBr, cm^{-1}) ν 3250, 2960, 1566. HRMS (ESI-ion trap) calcd. for $C_{19}H_{26}Cl_2N_{11}O_2$: $[M+H]^+$ 510.1642. Found: 510.1640.

2.4 Synthesis of 3b.



3b was obtained from flash column chromatographed on a silica gel column (petroleum ether and ethyl acetate = 5 / 1). (885.0 mg, 68% yield): white solid, mp 128-130 °C; ^{1}H NMR (400 MHz, $C_2D_2Cl_4$, 120°C): δ 7.66 (br, 2H), 7.42 (t, J = 7.8Hz, 1H), 6.70 (br, 2H), 6.23 (d, J = 7.8Hz, 2H), 5.15-5.09 (m, 2H), 1.74-1.58 (m, 8H), 1.33-1.28 (m, 16H), 0.90 (t, J = 6.8Hz, 12H); ^{13}C NMR (100 MHz, $C_2D_2Cl_4$, 120°C) δ 171.6, 171.4, 169.1, 158.0, 140.2, 90.1, 80.0, 33.6, 27.3, 22.5, 13.8; IR (KBr, cm^{-1}) v 3284, 2957, 2933, 2862, 1601, 1567, 1457. HRMS (ESI-ion trap) calcd. for $C_{29}H_{46}Cl_2N_{11}O_2$: $[M+H]^+$ 650.3208. Found: 650.3204.

2.4 Synthesis of 3c.

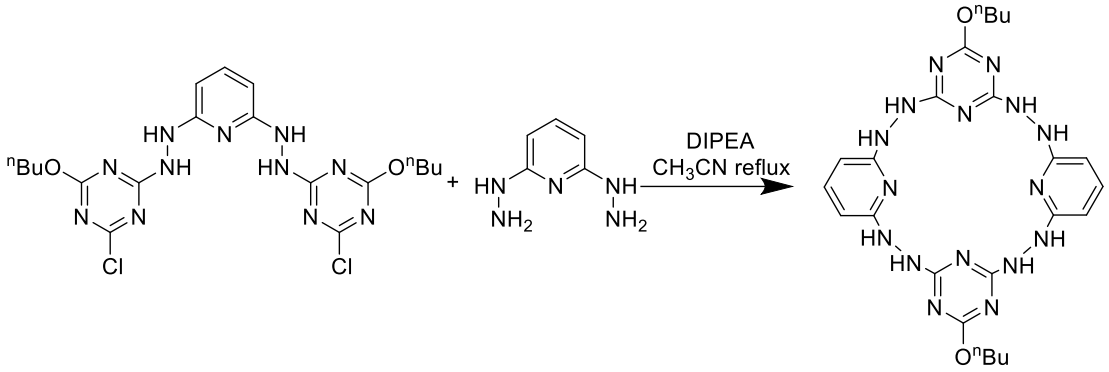


3c was obtained from flash column chromatographed on a silica gel column (petroleum ether and ethyl acetate = 1 / 3). (498.4.0 mg, 30%): pale yellow oil; IR (KBr, cm^{-1}) v 3262, 2928, 1727, 1566. HRMS (ESI-ion trap) calcd. for $C_{33}H_{49}Cl_2N_{11}O_{10}Na$: $[M+Na]^+$ 852.2933. Found: 852.2937.

It should be noted that the NMR spectra of these linear timer **3a-c** are complicated because of the rotation energy barrier of the N-N bond. ^{1}H NMR of trimers show several sets of signals at room temperature. The NMR spectra of **3a** and **3b** were then obtained

in $CDCl_2CDCl_2$ at 120 °C. Since **3c** was unstable at 120 °C. the attempts to obtain a meaningful NMR spectrum of **3c** failed.

2.4 Synthesis of 4a.



3a **1** **4a**

Both solutions of a mixture of 2,6-dihydrazinylpyridine **1** (2 mmol) and DIPEA (2.4 equiv) in acetonitrile (100 mL) and of the trimer **3a-c** (2 mmol) in acetonitrile (100 mL) were added dropwise at the same time and the same rate to a refluxing acetonitrile (200 mL). After addition of two reactants, the resulting mixture was stirred for another 8 h until the starting materials were consumed. Pale yellow precipitate formed during the reaction was collected, and the solid mixture of crude products contained the desired all hydrazo-bridged calix[2]pyridine[2]triazine **4a** and its lager ring homolog calix[4]pyridine[4]triazine, as supported by HRMS and ^{1}H NMR spectra, in the ratio of 4 to1 (Figure S1-2). Recrystallization of the crude products twice from a mixture of DMSO and acetonitrile gave pure **4a**. The lager ring homolog calix[4]pyridine[4]triazine was not obtained in pure form.

4a (484 mg, 42% yield): white solid, mp >300 °C; ^{1}H NMR (400 MHz, DMSO, 60°C) δ 8.98 (s, 4H), 8.65 (br, 4H), 6.98 (t, J = 8.0Hz, 2H), 5.48 (d, J = 7.8Hz, 4H), 4.25 (t, J = 6.4Hz, 4H), 1.71-1.64 (m, 4H), 1.48-1.38 (m, 4H), 0.95 (t, J = 7.8Hz, 6H); ^{13}C NMR (100 MHz, DMSO, 60°C) δ 170.1, 169.1, 158.5, 138.5, 94.3, 65.6, 30.3, 18.4, 13.3; IR (KBr, cm^{-1}) ν 3229, 2960, 1589, 1419. HRMS (ESI-ion trap) calcd. for $C_{24}H_{33}N_{16}O_2$: $[M+H]^+$ 577.2967. Found: 577.2968. Elemental analysis calcd. (%) for $C_{24}H_{32}N_{16}O_2$: C, 49.99; H, 5.59; N, 38.87. Found: C, 49.84; H, 5.67; N, 38.82.

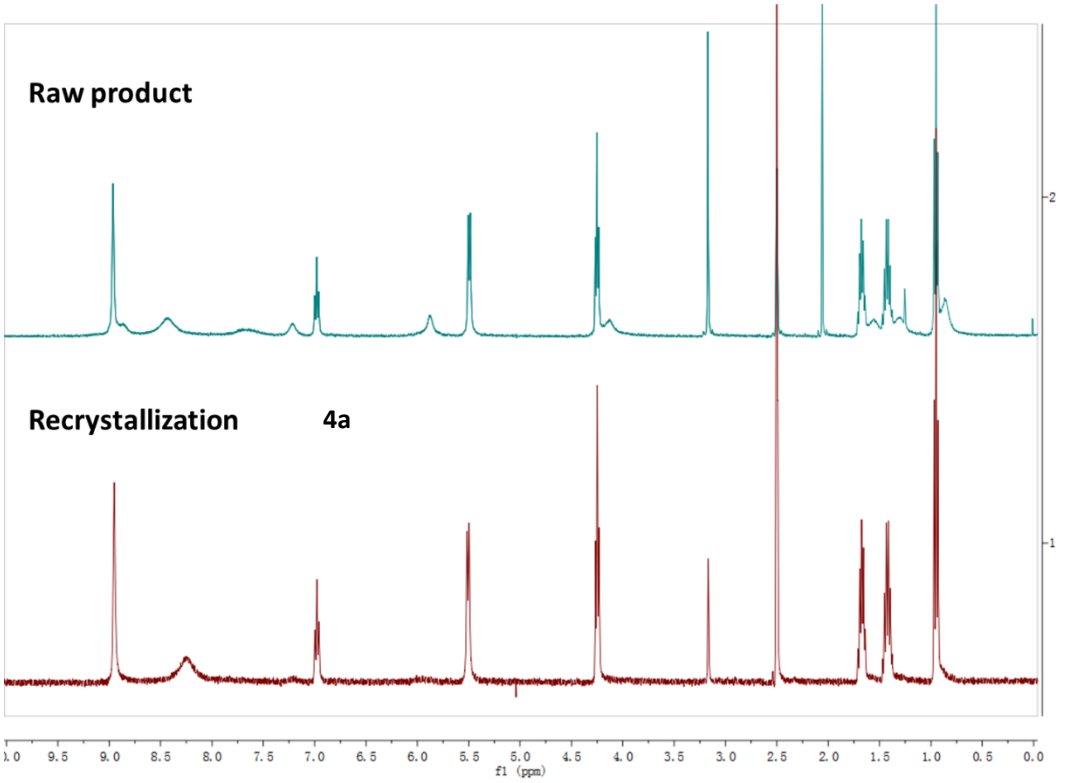
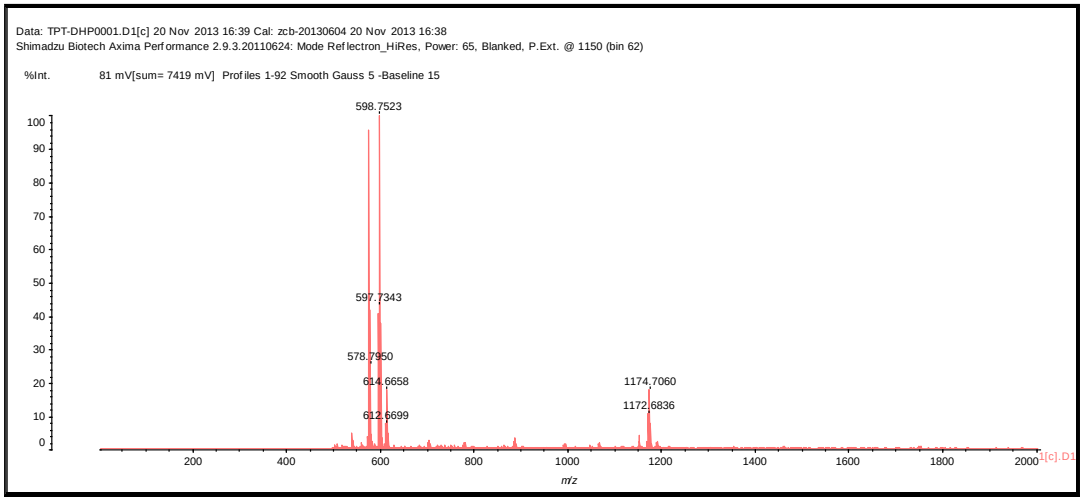
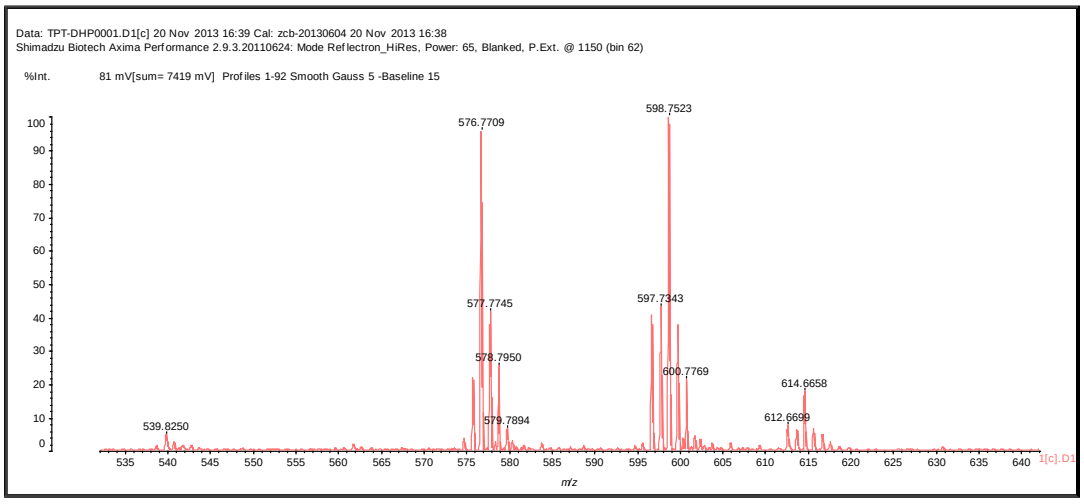


Figure S1. ^{1}H NMR spectra of crude product and **4a**

a)



b)



c)

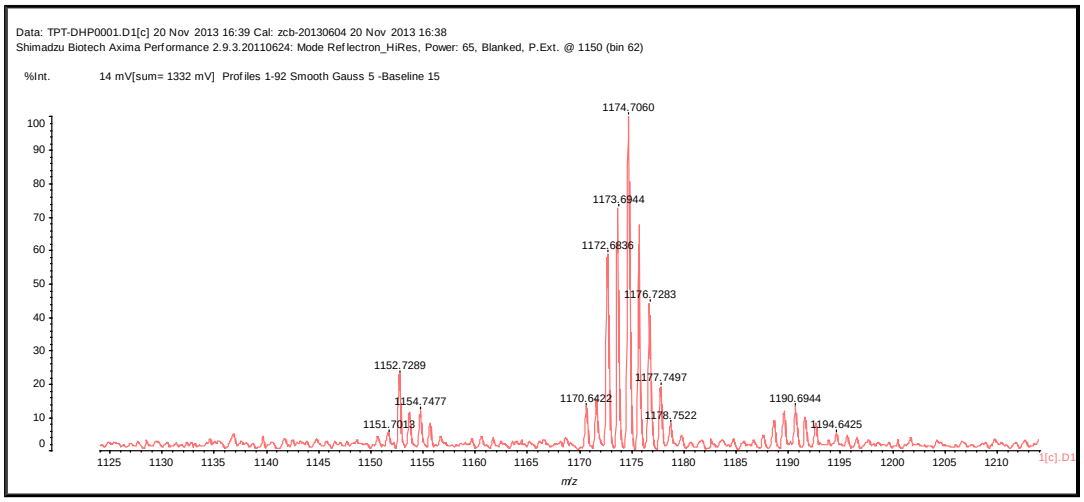
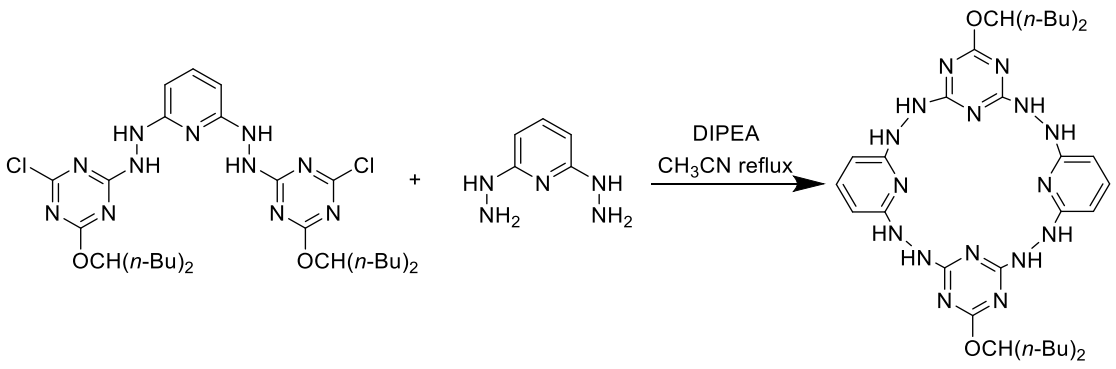


Figure S2. a) MALDI-TOF HRMS of the crude product; b) Partial expansion of the spectrum. c) Partial expansion of the spectrum.

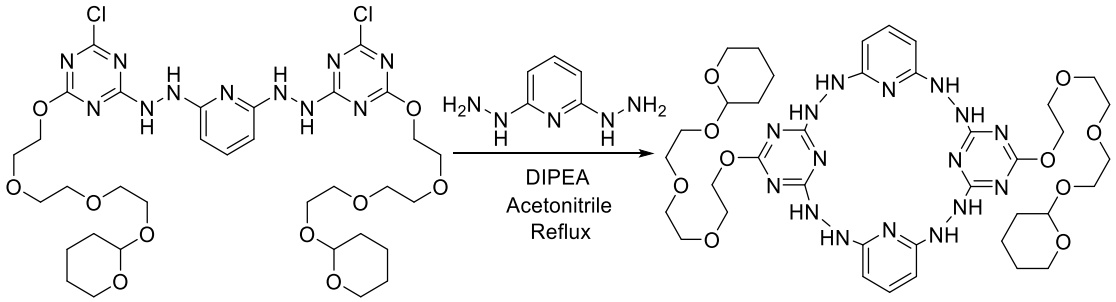
2.5 Synthesis of 4b.



3b **1** **4b**

4b was obtained from flash column chromatographed on a silica gel column ($CHCl_3$ / methanol = 20 / 1) (501 mg, 35% yield): white solid, mp 200-202 ºC; ^{1}H NMR (400 MHz, DMSO, 60ºC) δ 8.89 (s, 4H), 8.27 (br, 4H), 6.99 (t, J = 7.8Hz, 2H), 5.51 (d, J = 7.8Hz, 4H), 5.13-5.07 (m, 2H), 1.64-1.58 (m, 8H), 1.37-1.30 (m, 16H), 0.92-0.88 (m, 12H); ^{13}C NMR (100 MHz, DMSO, 60ºC) δ 170.1, 169.0, 158.6, 138.4, 94.4, 75.0, 32.9, 26.6, 21.9, 13.5; IR (KBr, cm^{-1}) ν 3286, 2955, 2869, 1571, 1414. HRMS (ESI-ion trap) calcd. for $C_{34}H_{53}N_{16}O_2$: $[M+H]^+$ 717.4532. Found: 717.4529. Elemental analysis calcd. (%) for $C_{34}H_{52}N_{16}O_2$: C, 56.96; H, 7.31; N, 31.26. Found: C, 56.59; H, 7.15; N, 31.05.

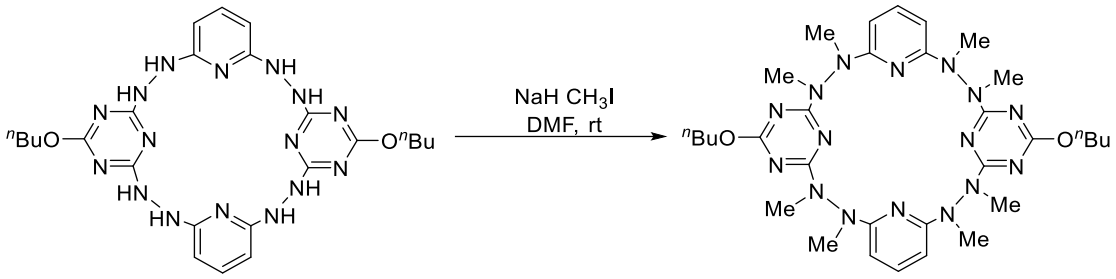
2.6 Synthesis of 4c.



3c **4c**

4c was obtained from recrystallization from a mixture of DMSO and DCM (574 mg, 32% yield): white solid, mp 259-260 ºC; ^{1}H NMR (400 MHz, DMSO, 100ºC) δ 8.79 (br, 4H), 7.63 (br, 4H), 7.00 (br, 2H), 5.62 (br, 4H), 4.58-4.61 (m, 2H), 4.37 (s, 4H), 3.80-3.40 (m, 24H), 1.80-1.45 (m, 12H); ^{13}C NMR (100 MHz, DMSO, 100ºC) δ 169.9, 168.9, 158.3, 138.2, 97.9, 94.4, 69.6, 69.5, 69.5, 68.3, 65.7, 65.1, 61.0, 29.9, 24.6, 18.7; IR (KBr, cm^{-1}) v 3290, 2942, 1575, 1416, 1334. HRMS (ESI-ion trap) calcd. for $C_{38}H_{56}N_{16}O_{10}Na$: $[M+Na]^+$ 919.4258. Found: 919.4267. Elemental analysis calcd. (%) for $C_{38}H_{56}N_{16}O_{10}$: C, 50.88; H, 6.29; N, 24.99. Found: C, 50.66; H, 6.29; N, 24.80.

2.7 Synthesis of 5.



4a **5**

An oven-dried Schlenk tube was charged with **4a** (1 mmol, 577mg) and NaH (12 mmol, 288mg). Under the protection of argon, the tube was cooled to 0° C in an ice-bath. DMSO (5.0 mL, deoxygenated prior to use) was added. The Schlenk tube was sealed and the reaction mixture was stirred at room temperature for 1 h. MeI (12 mmol, 1.7g) was added dropwise and the reaction mixture was stirred overnight. The reaction was quenched by adding a dilute hydrochloric acid (1 N). The resulting suspension was extracted with ethyl acetate (3 x 50 mL) and then dried with anhydrous Na_2SO_4. After removal of solvent, the residue was chromatographed on a silica gel column with a mixture of petroleum ether and ethyl acetate (5:1) as the mobile phase to give pure linear trimer **5.**

5 (586 mg, 85% yield): white solid, mp 257-258 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.84 (t, J = 8.0Hz, 2H), 5.38 (d, J = 8.0Hz, 4H), 4.36 (t, J = 6.8Hz, 4H), 3.32 (s, 12H), 3.29 (s, 12H), 1.82-1.64 (m, 4H), 1.53-1.47 (m, 4H), 0.99 (t, J = 7.2Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.3, 167.9, 156.9, 138.6, 94.2, 67.0, 34.6, 34.4, 31.1, 19.4, 14.0; IR (KBr, cm^{-1}) ν 2954, 1932, 2870, 1592, 1561, 1538. HRMS (ESI-ion trap) calcd. for $C_{32}H_{49}N_{16}O_2$: $[M+H]^+$ 689.4219. Found: 689.4216. Elemental analysis calcd. (%) for $C_{32}H_{48}N_{16}O_2$: C, 55.80; H, 7.02; N, 32.53. Found: C, 55.82; H, 7.07; N, 32.32.

3. Crystal Structures

Compound	**4a**	**5**	**5**·$[(Co(ClO_4)_2(H_2O)_3]_2$
empirical formula	$C_{22}H_{10}Cl_4F_8N_6O_6$	C_{34} H_{52} Cl_4 N_{16} O_2	C_{36} H_{60} Co_2 N_{16} O_{24}
M_r	1387.69	858.71	1312.62
crystal size [mm^3]	0.53×0.38×0.19	0.51 x 0.37 x 0.22	0.10 x 0.08 x 0.04
crystal system	triclinic	triclinic	monoclinic
space group	P-1	P-1	$P\,2_1/c$
a [Å]	13.180(3)	9.8942(18)	11.9766(4)
b [Å]	19.895(4)	11.644(2)	19.3355(5)
c [Å]	20.590(4)	20.487(4)	12.7804(4)
α [deg]	108.27(3)	87.078(8)	90
β [deg]	107.63(3)	78.302(9)	114.734(4)
γ [deg]	90.48(3)	68.651(6)	90
V [$Å^3$]	4854(2)	2151.9(8)	2688.08(17)
d [g/cm^3]	0.949	1.325	1.622
Z	2	2	12
T [K]	173.1500	173.1500	173.00(10)
R factor [I>2σ(I)]	0.1520	0.1118	0.0672
R factor (all data)	0.1775	0.1208	0.073
quality of fit	1.136	1.120	1.048

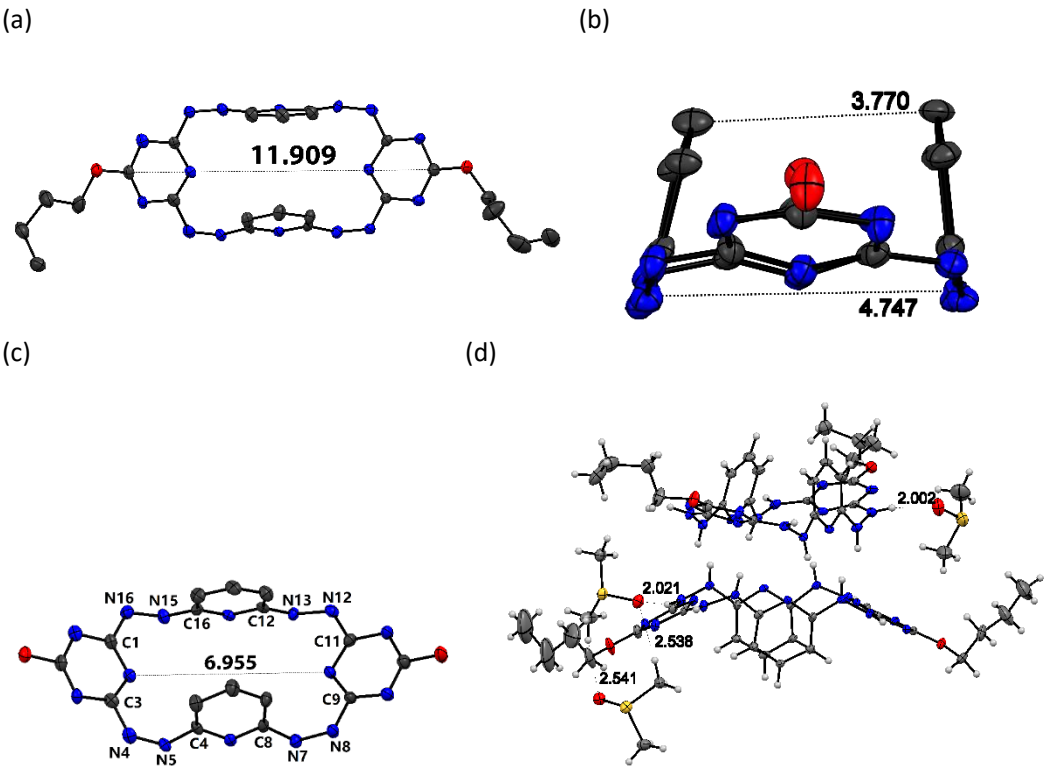


Figure S6 X-ray molecular structure of **4a.** (a) top view; (b) side views; (c) side views with partial atom labeling and (d) **4a**·3DMSO in side views. Alkyl substituents were omitted for clarity in (b), (c) structures. Selected bond lengths (Å): N4-C3 1.386(9), N5-C4 1.371(7), N7-C8 1.390(8), N8-C9 1.358(7), N12-C11 1.332(8), N13-C12 1.386(7), N15-C16 1.370(7), N16-C1 1.359(8); Selected bond

angles(°): N5-N4-C3 123.6(5), N4-N5-C4 120.7(5), N8-N7-C8 121.8(5), C9-N8-N7 123.1(5), C11-N12-N13 122.9(5), C12-N13-N12 119.7(4), C16-N15-N16 122.0(5), C1-N16-N15 121.0(5).

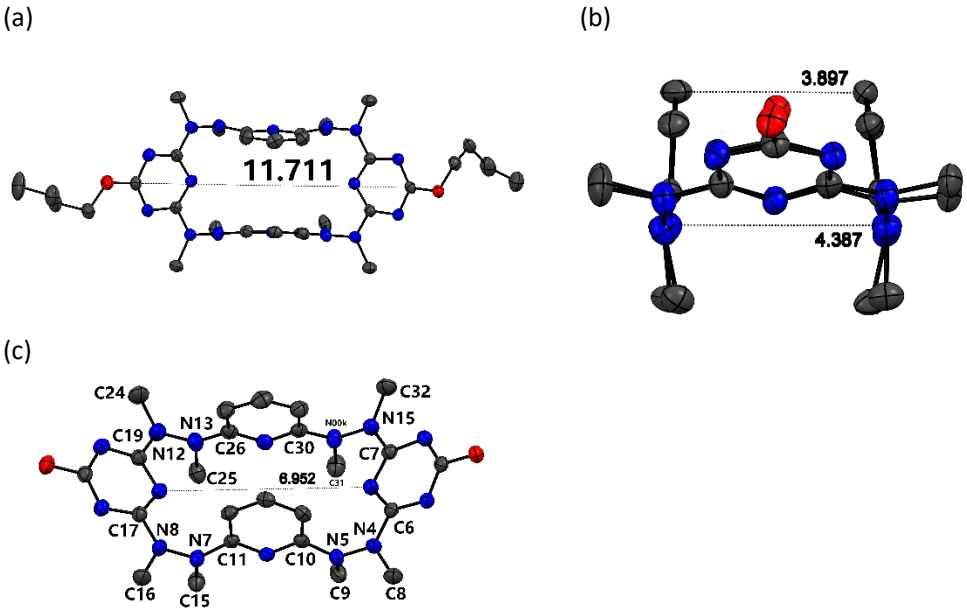
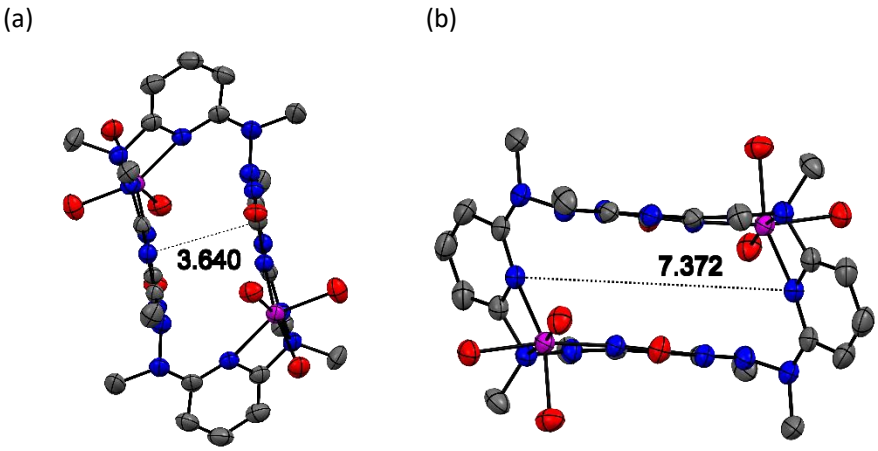


Figure S7 X-ray molecular structure of **5.** (a) top view; (b) side views; (c) side views with partial atom labeling. Alkyl substituents were omitted for clarity in side-view structures. Selected bond lengths (Å): N4-C6 1.359(4), N4-C8 1.448(5), N5-C9 1.463(5), N5-C10 1.394(4), N7-C11 1.387(4), N7-C15 1.441(5), N8-C16 1.459(4), N8-C17 1.366(4), N12-C19 1.361(4), N12-C24 1.461(5), N13-C25 1.449(5), N13-C26 1.389(4), N15-C7 1.358(4), N15-C32 1.460(4), N00K-C30 1.385(4), N00K-C31 1.449(5); Selected bond angles: N5-N4-C8 115.7(3), C6-N4-N5 120.1(3), C6-N4-C8 123.9(3), N4-N5-C9 115.8(3), N4-N5-C10 117.8(3), C10-N5-C9 122.6(3), N8-N7-C11 118.6(3), N8-N7-C15 116.4(3), C11-N7-C15 124.3(3), N7-N8-C16 115.4(3), C17-N8-N7 119.7(3), C17-N8-C16 124.5(3), N13-N12-C24 116.4(3), C19-N12-N13 120.5(3), C19-N12-C24 123.1(3), N12-N13-C25 117.7(3), N12-N13-C26 117.2(3), C26-N13-C25 122.0(3), N00K-N15-C32 115.9(3), C7-N15-N00K 119.7(3), C7-N15-C32 124.4(3), N15-N00K-C31 117.0(3), C30-N00K-N15 117.7(3), C30-N00K-C31 124.1(3).



(c)

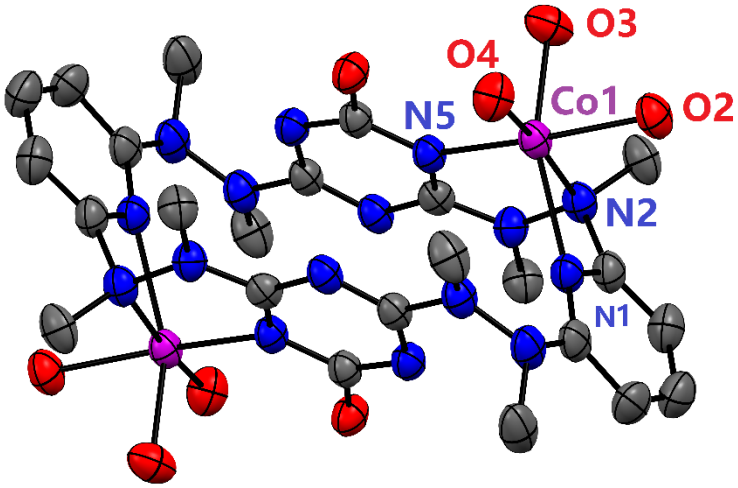


Figure S8 X-ray molecular structure of **5**·$[(Co(ClO_4)_2(H_2O)_3]_2$. (a) top view; (b) side views; (c) side views with partial atom labeling. Alkyl substituents and free perchlorate groups were omitted for clarity. Selected bond lengths (Å): Co1-O2 2.121(3), Co1-O3 2.084(3), Co1-O4 2.038(3), Co1-N1 2.255(3), Co1-N2 2.191(3), Co1-N5 2.061(3).

4. NMR Titration

Stacked ^{1}H NMR spectra of **4a** in d_6-DMSO with the increase of concentration (6, 8, 20, 40, 60, 80 mM) at 333K (Figure S9).

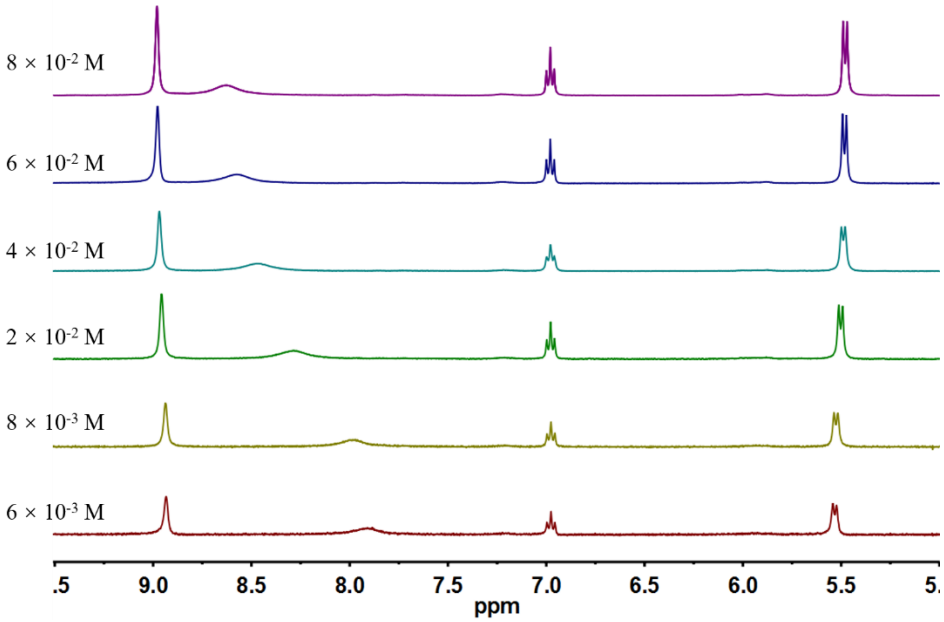


Figure S9 Stacked ^{1}H NMR spectra of **4a** in d_6-DMSO at 60 °C

Stacked ^{1}H NMR spectra of **4b** in $Cl_2CDCDCl_2$ with the increase of concentration (6, 8, 20, 40, 60, 80 mM) at 333K (Figure S10).

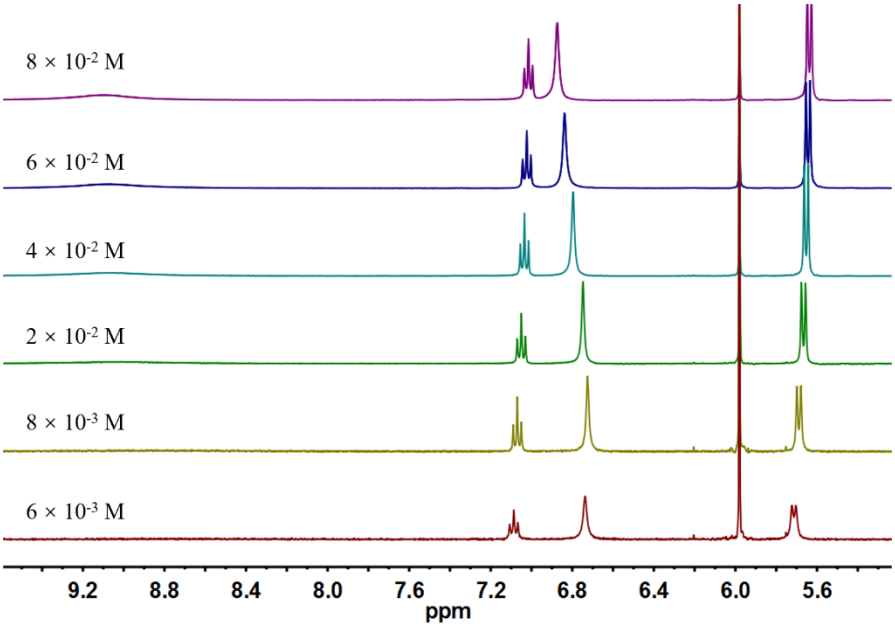


Figure S10 Stacked ^{1}H NMR spectra of **4b** in $CDCl_2CDCl_2$ at 60 °C

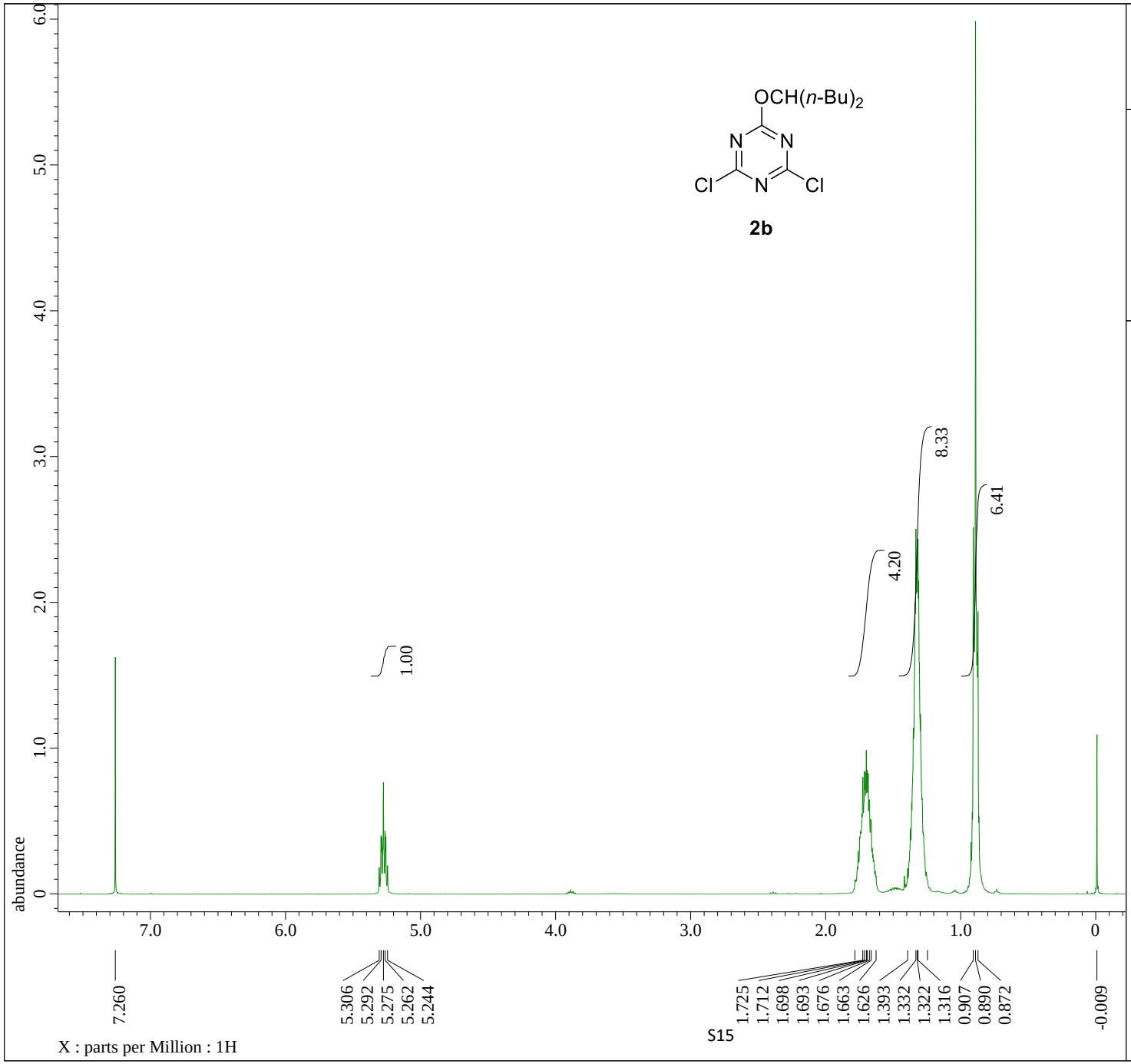
5. References

[1] Brien, K. A.; Garner, C. M.; Klausmeyer, K. K.; Feazell, R. P. *J. Chem. Crystallogr.* **2005,** *35*, 875-883.

[2] Naseer, M. M.; Wang, D.-X.; Zhao, L.; Huang, Z.-T.; Wang, M.-X. *J. Org. Chem.* **2011,** *76*, 1804-1813.

[3] Richard, A.; Bourel-Bonnet, L. *Chem. Eur. J.* **2005,** *11*, 7315-7321.

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

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ppm
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X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
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Tri_Offset        = 5[ppm]
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Total_Scans       = 8

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X_90_Width        = 12[us]
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X_Angle           = 45[deg]
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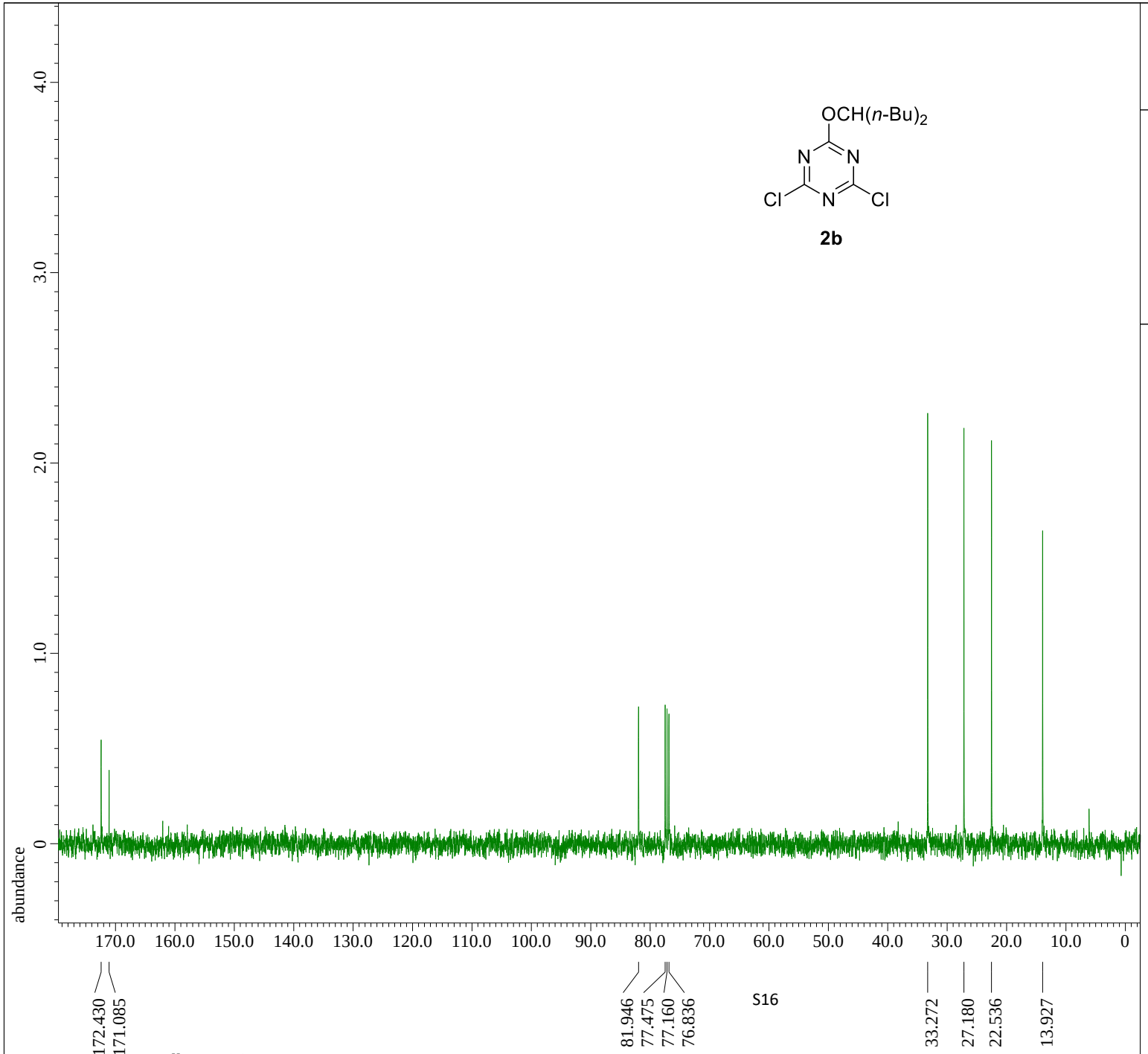

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X_Freq	= 100.52530333[MHz]
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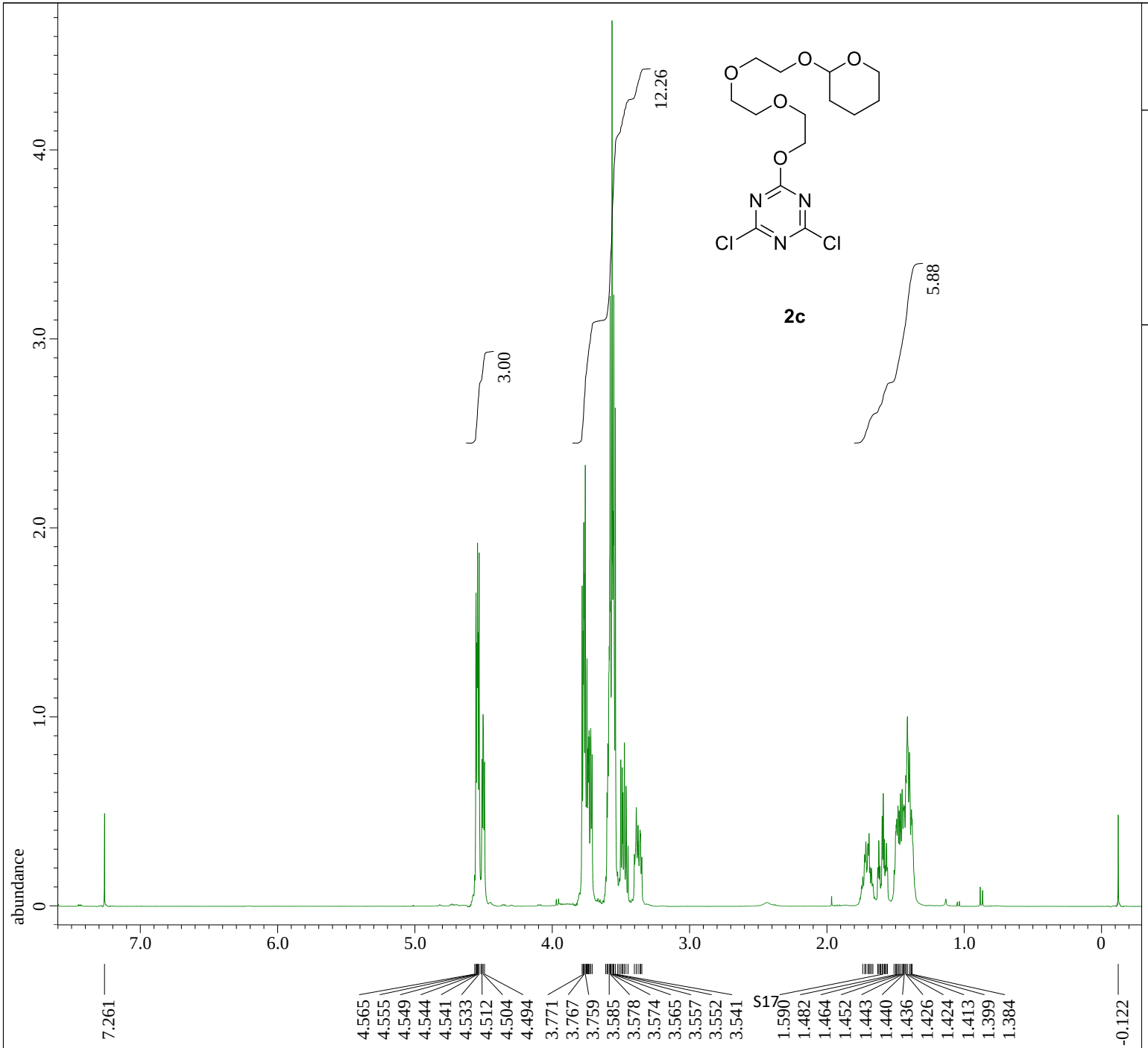


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ppm
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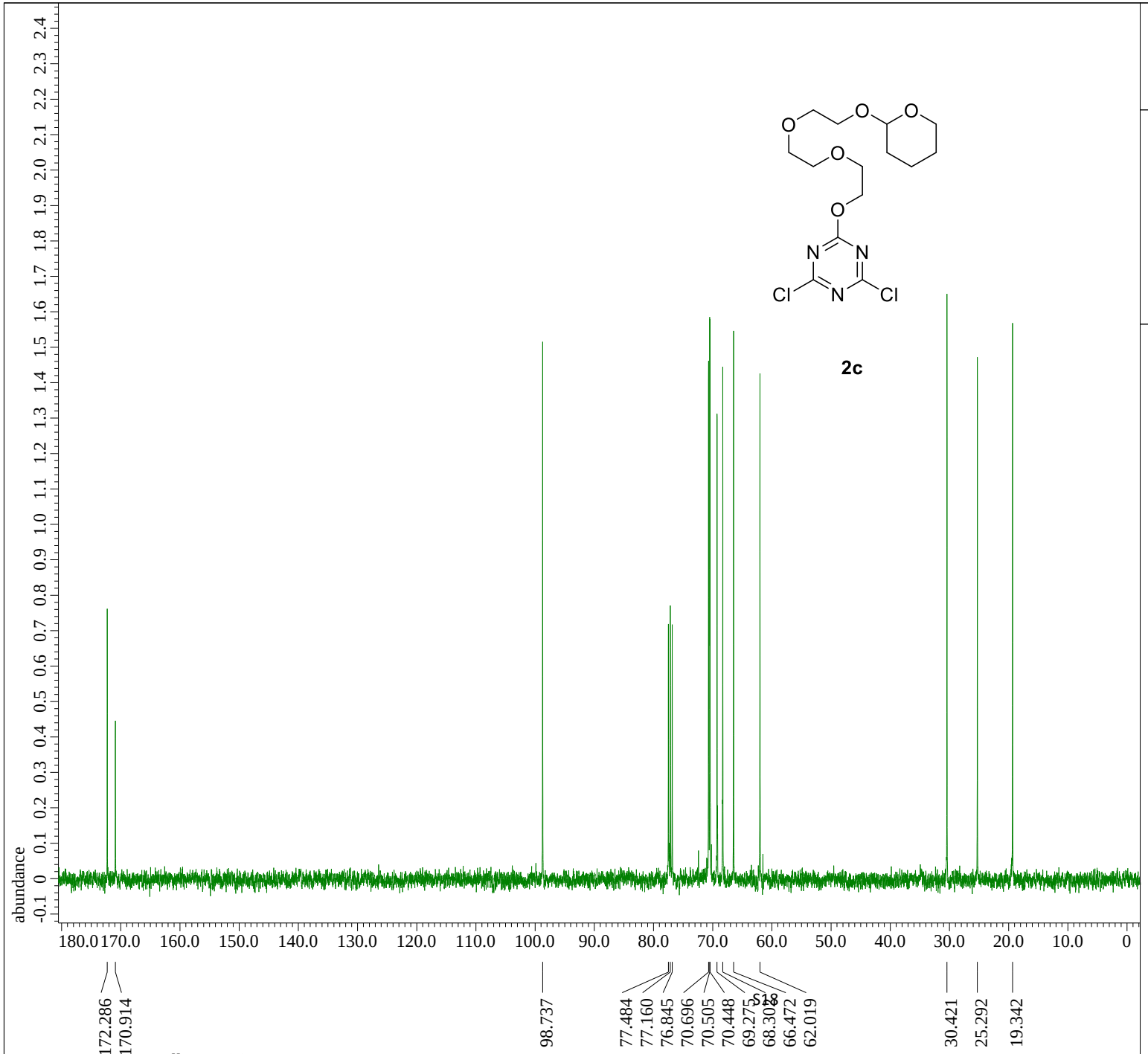

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X_Offset	= 100[ppm]
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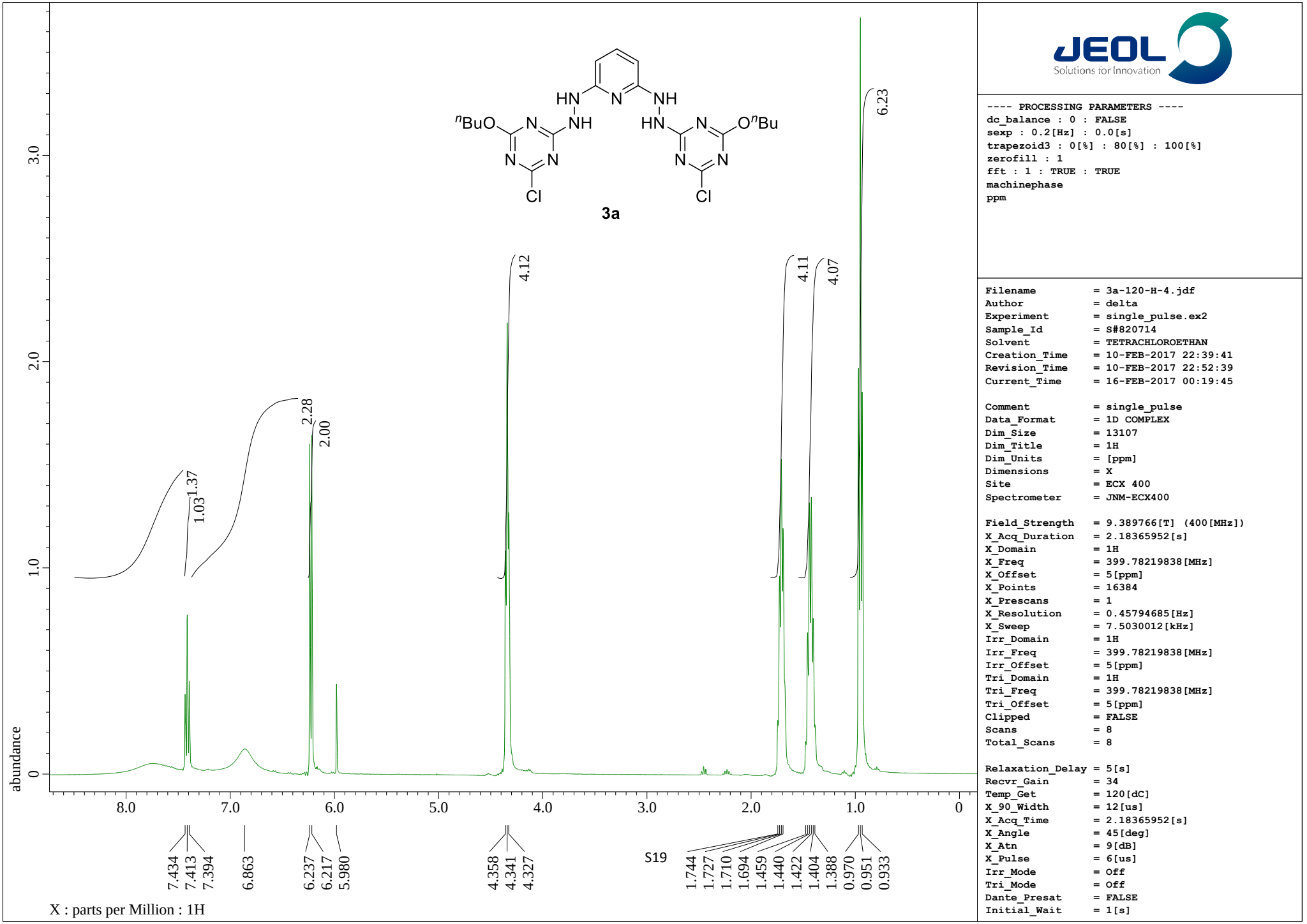

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Scans	= 8
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Dante_Presat	= FALSE
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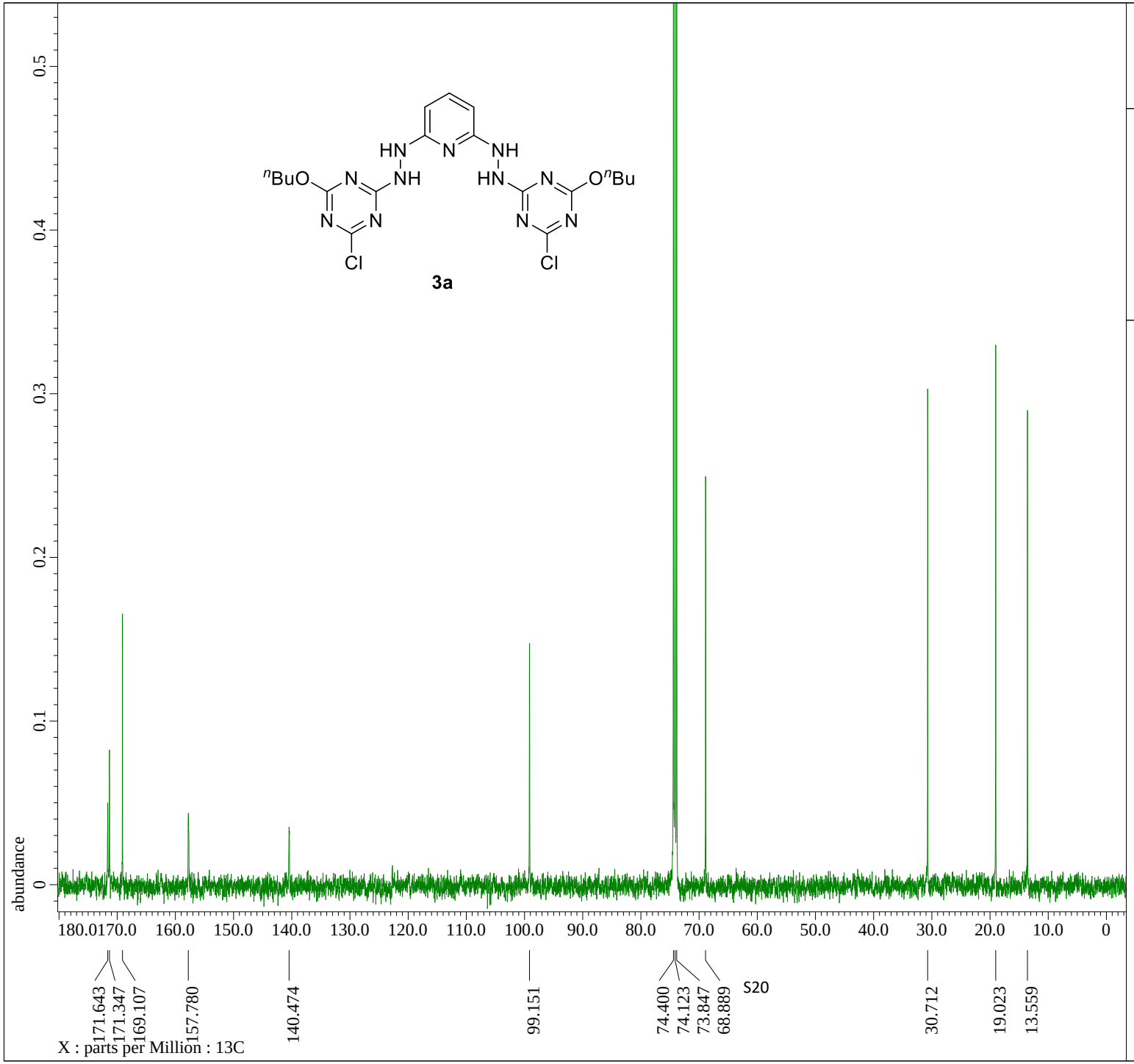

3a

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Irr_Atn_Noe       = 23.03[dB]
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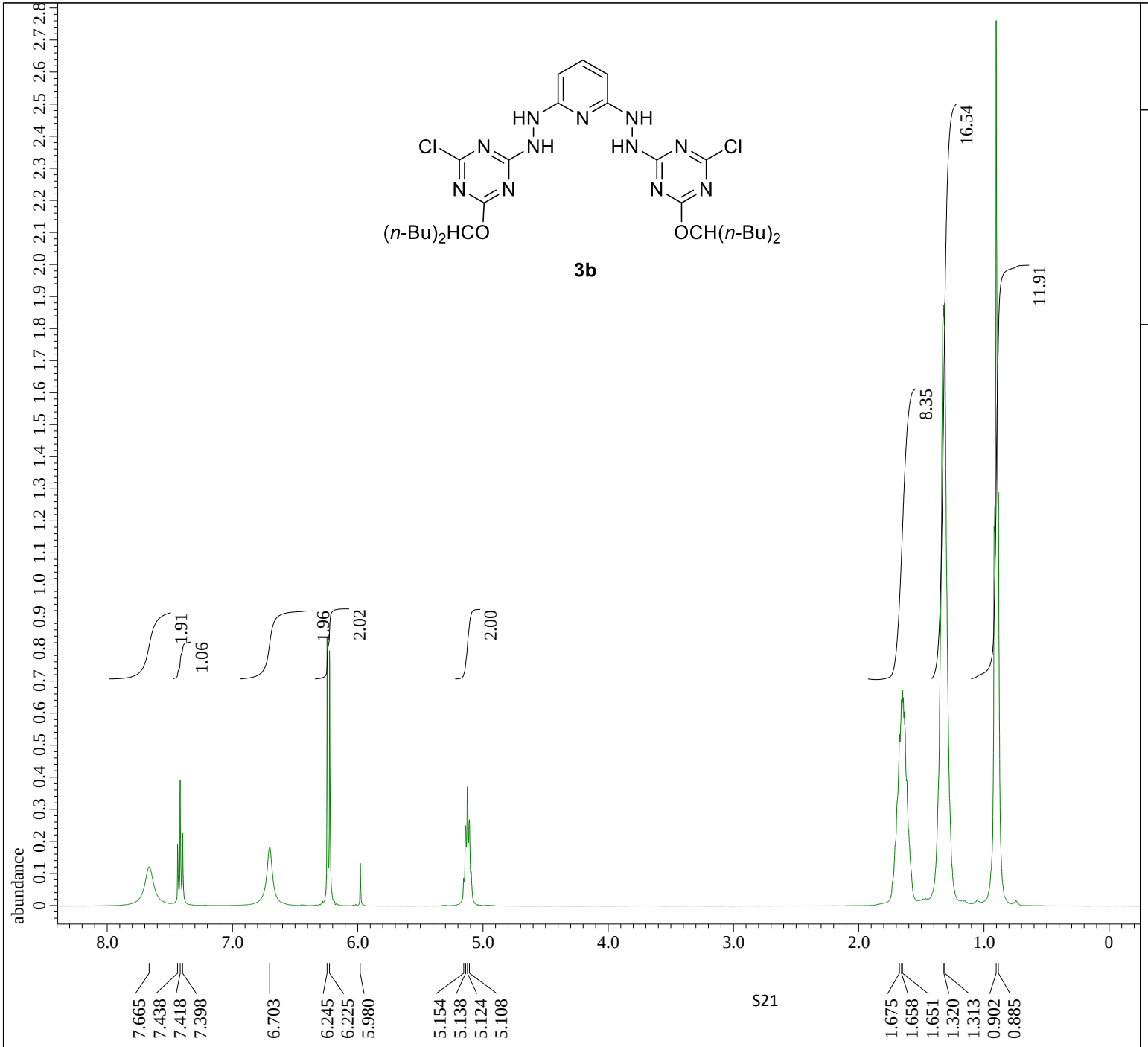


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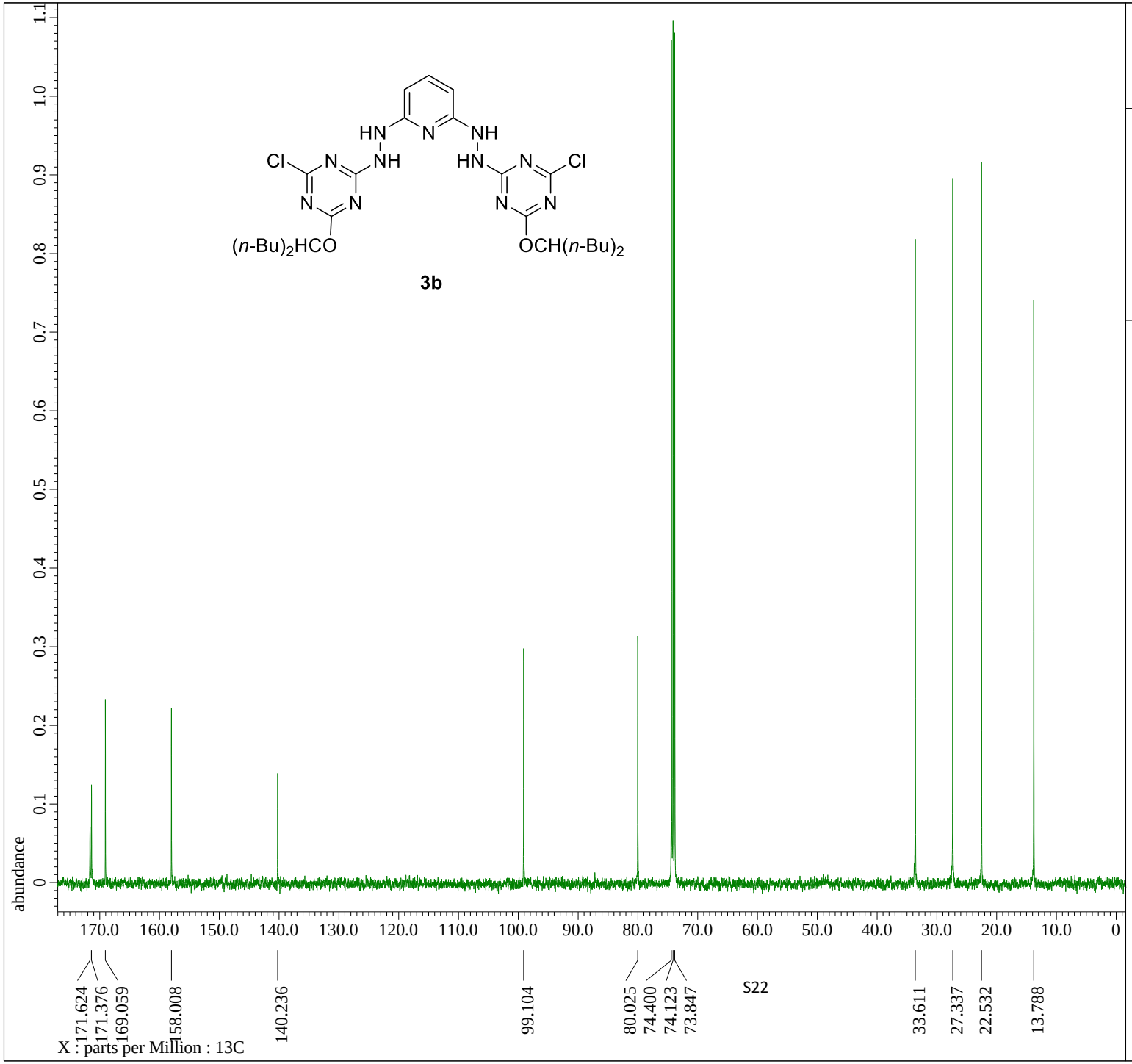

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X_Sweep           = 31.40703518[kHz]
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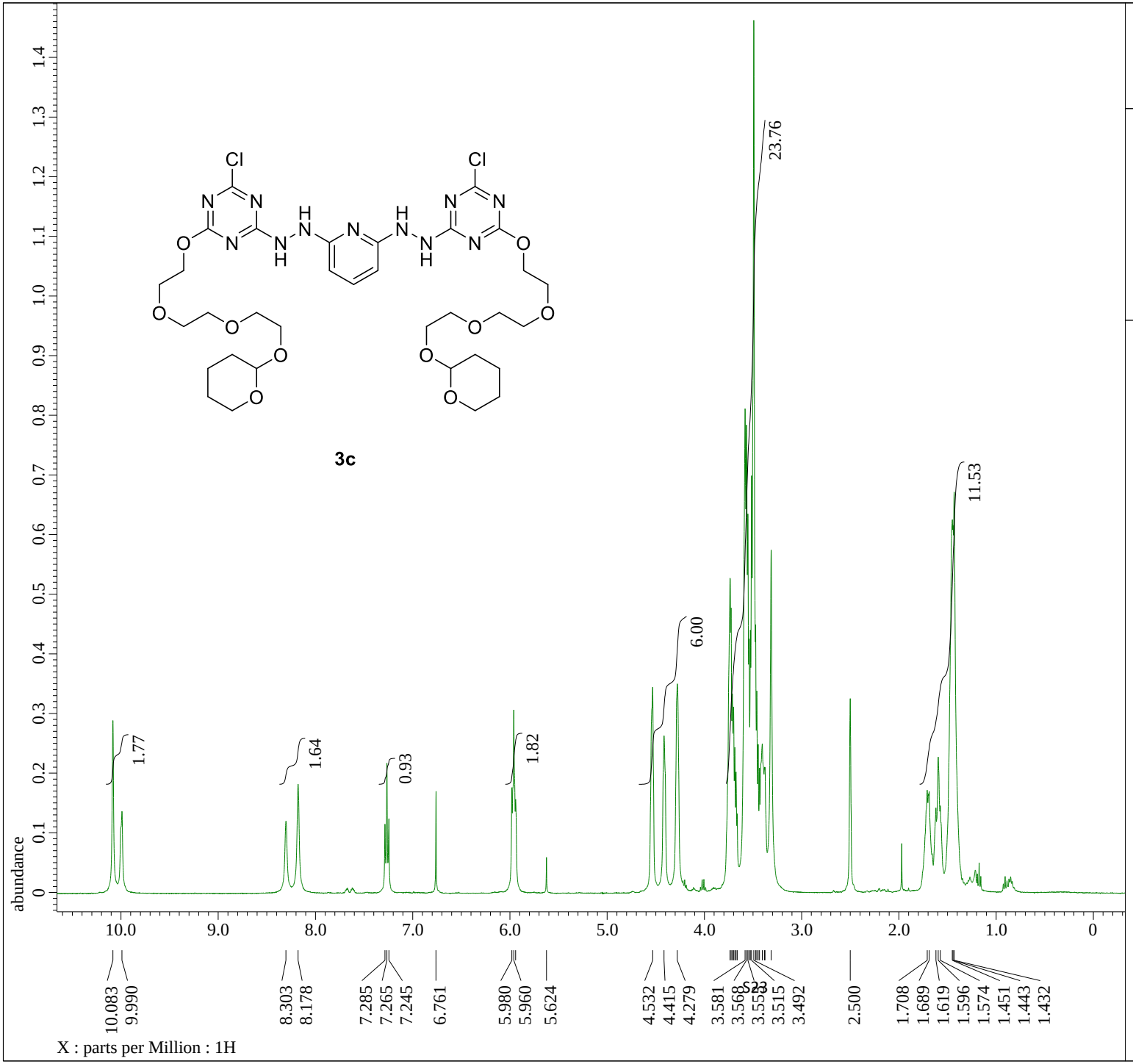

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X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
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X_Resolution	= 0.45794685[Hz]
X_Sweep	= 7.5030012[kHz]
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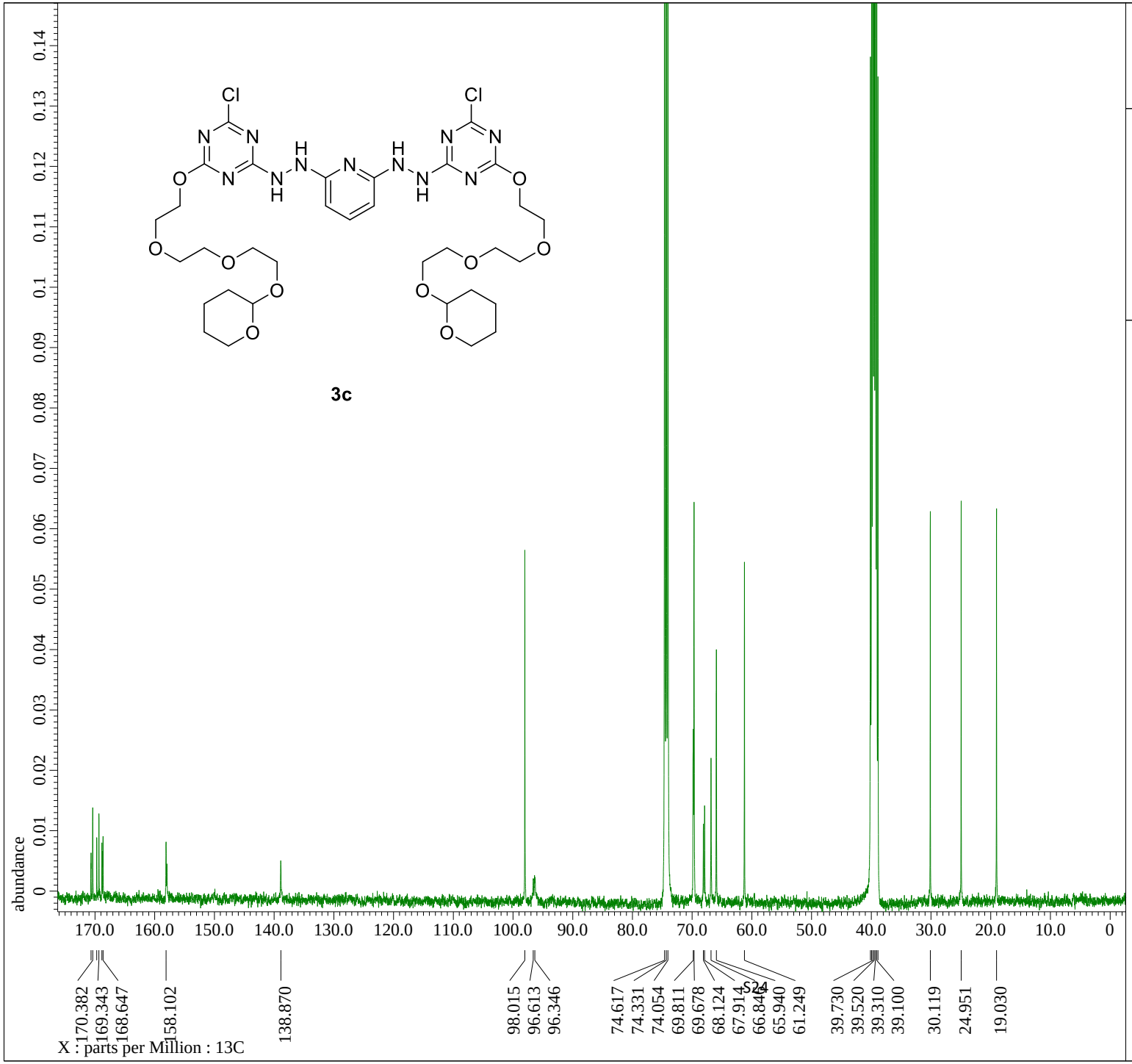

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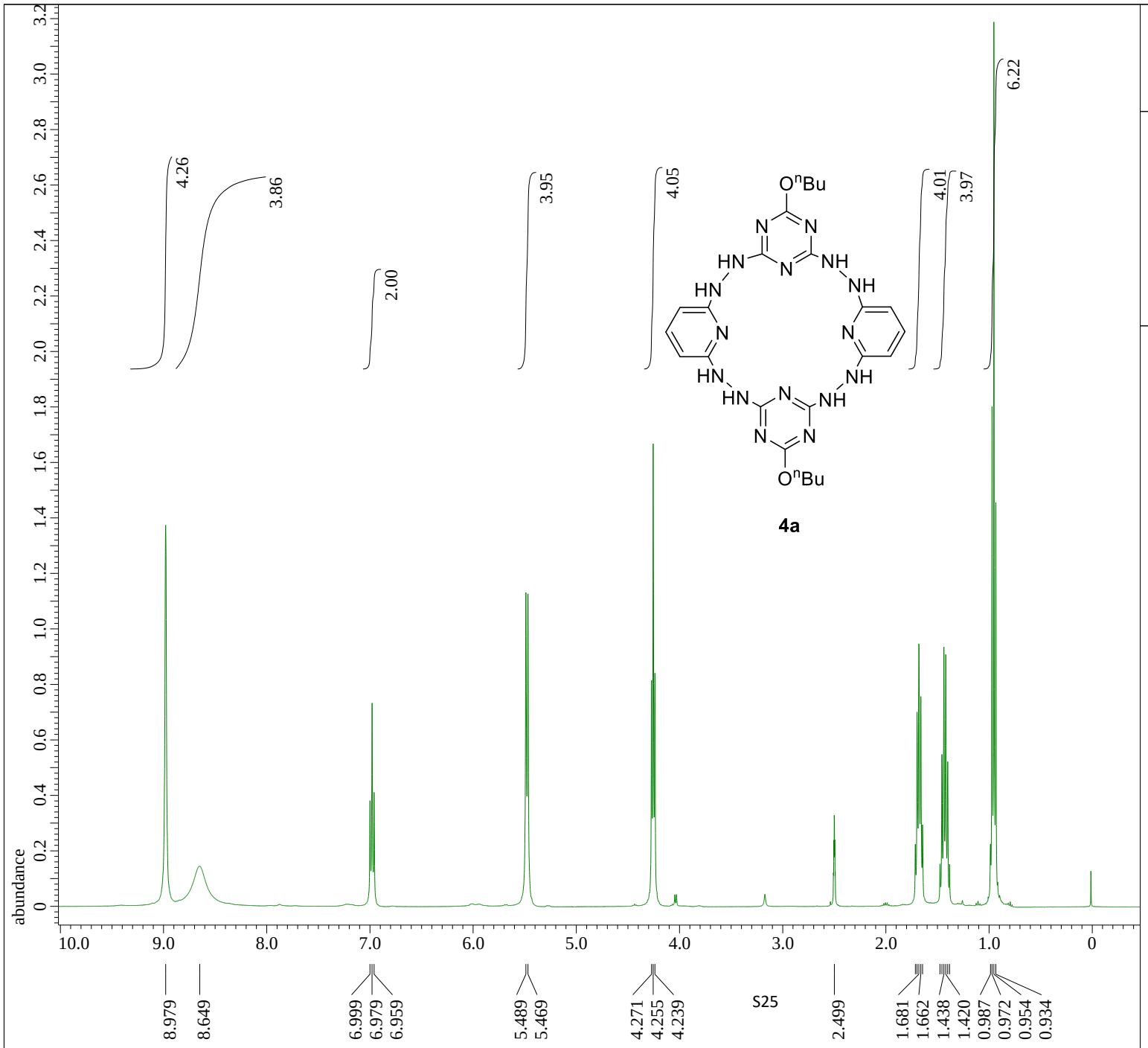
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

Filename	= 20170214-3c-DMSO-C-3.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= S#32220
Solvent	= DMSO-D6
Creation_Time	= 14-FEB-2017 09:13:38
Revision_Time	= 14-FEB-2017 16:04:30
Current_Time	= 16-FEB-2017 00:49:25
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= JNM-ECX400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= TRUE
Scans	= 10000
Total_Scans	= 10000
Relaxation_Delay	= 2[s]
Recvr_Gain	= 54
Temp_Get	= 18.4[dC]
X_90_Width	= 9[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 8.823[dB]
X_Pulse	= 3[us]
Irr_Atn_Dec	= 23.03[dB]
Irr_Atn_Noe	= 23.03[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]



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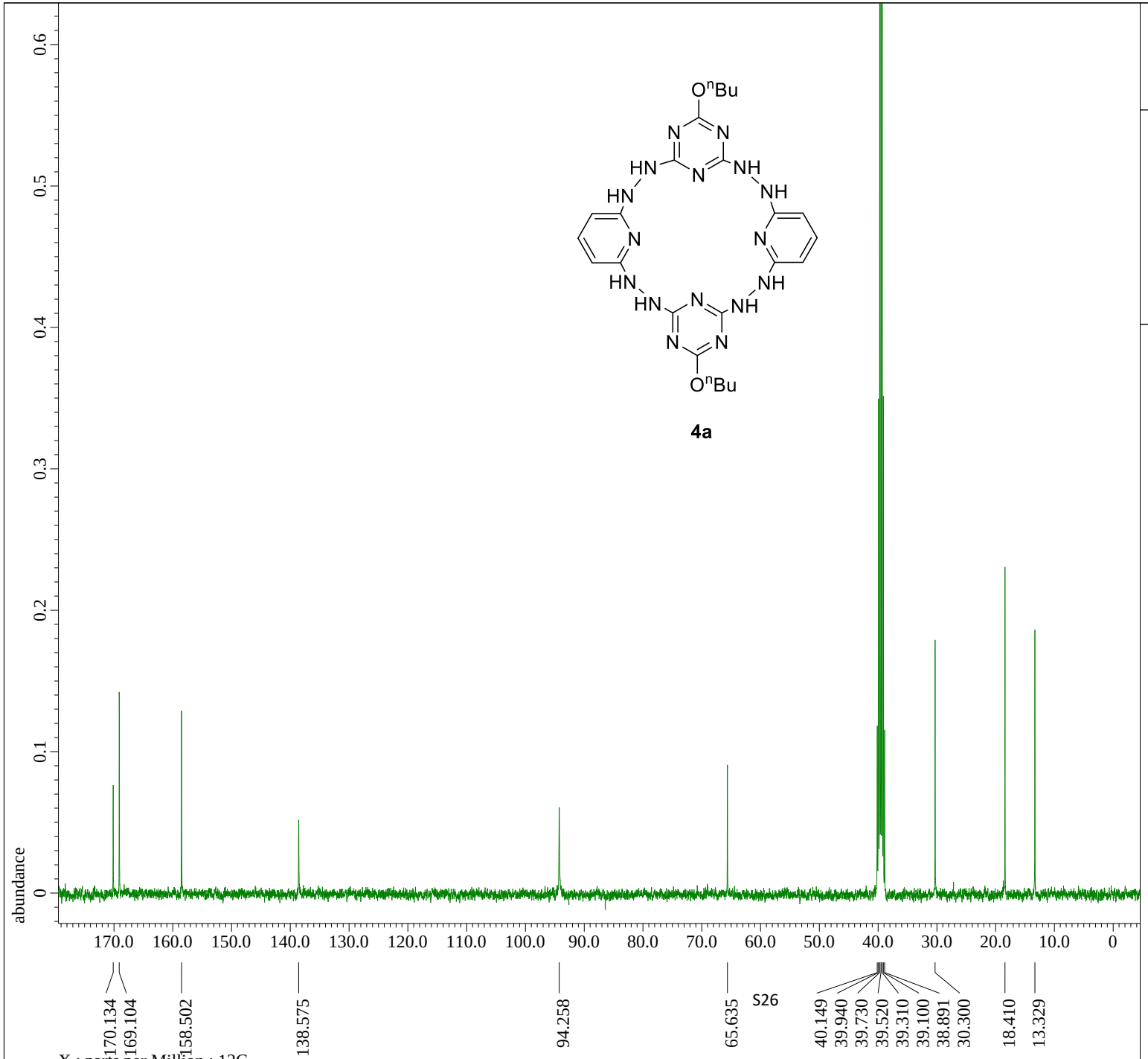
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

Filename	= 4a-H-3.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_Id	= S#150361
Solvent	= DMSO-D6
Creation_Time	= 8-FEB-2017 04:02:48
Revision_Time	= 8-FEB-2017 04:15:47
Current_Time	= 16-FEB-2017 00:22:58
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= 1H
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= JNM-ECX400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 2.18365952[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.45794685[Hz]
X_Sweep	= 7.5030012[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 1H
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 30
Temp_Get	= 60[dC]
X_90_Width	= 12[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 3.4[dB]
X_Pulse	= 6[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]



4a

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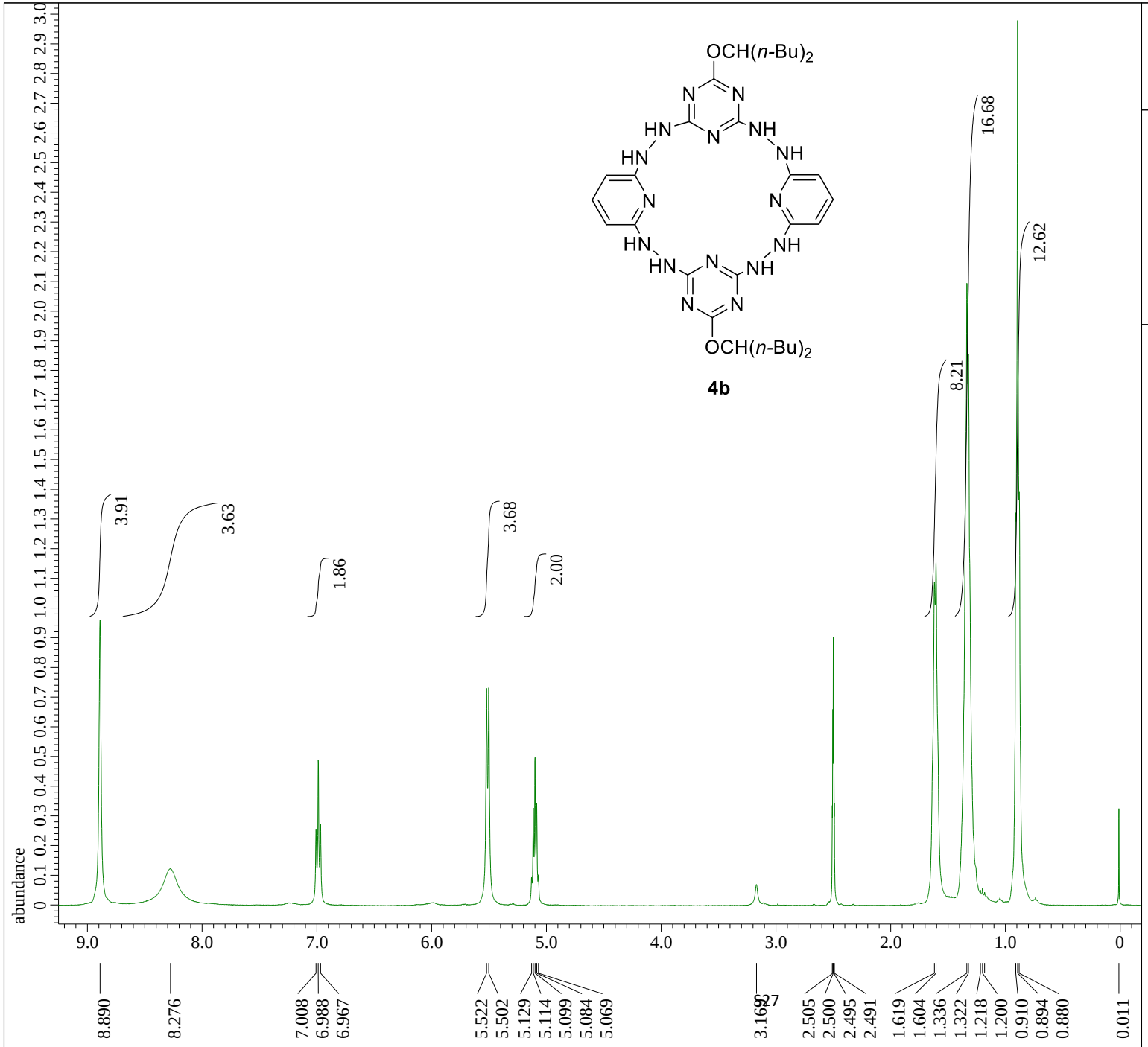
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

Filename	= 4a-C-3.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= S#136608
Solvent	= DMSO-D6
Creation_Time	= 8-FEB-2017 04:00:57
Revision_Time	= 8-FEB-2017 04:10:53
Current_Time	= 16-FEB-2017 00:23:32
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= JNM-ECX400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 400
Total_Scans	= 400
Relaxation_Delay	= 2[s]
Recvr_Gain	= 52
Temp_Get	= 60[dC]
X_90_Width	= 9[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 8.823[dB]
X_Pulse	= 3[us]
Irr_Atn_Dec	= 23.03[dB]
Irr_Atn_Noe	= 23.03[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]



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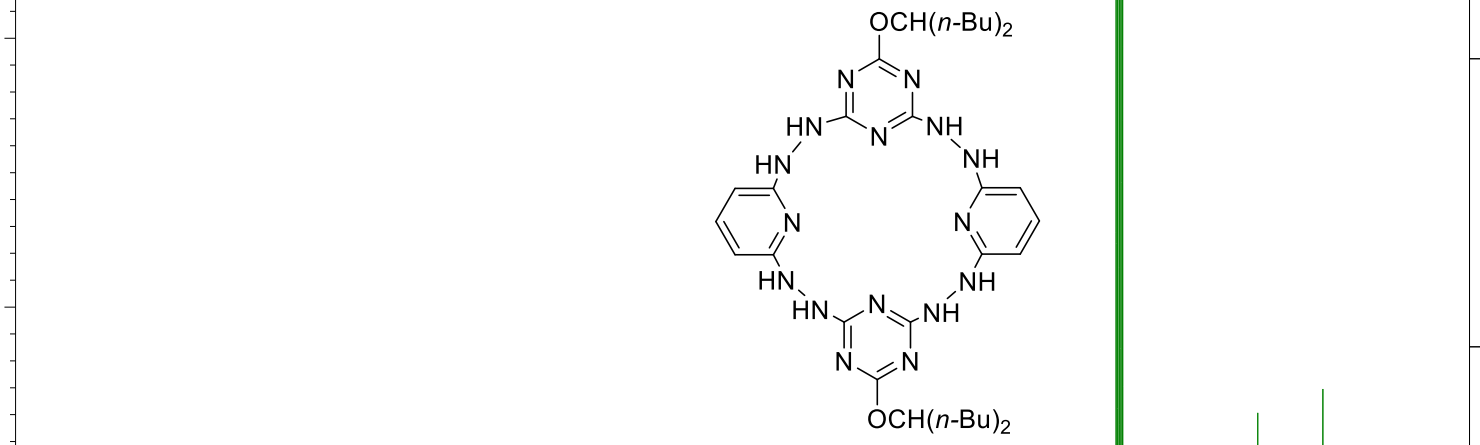
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = 4b-H-5.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#110800
Solvent           = DMSO-D6
Creation_Time     =  8-FEB-2017 02:56:52
Revision_Time     =  8-FEB-2017 05:02:51
Current_Time      = 16-FEB-2017 00:24:10

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 44
Temp_Get          = 60[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```



4b

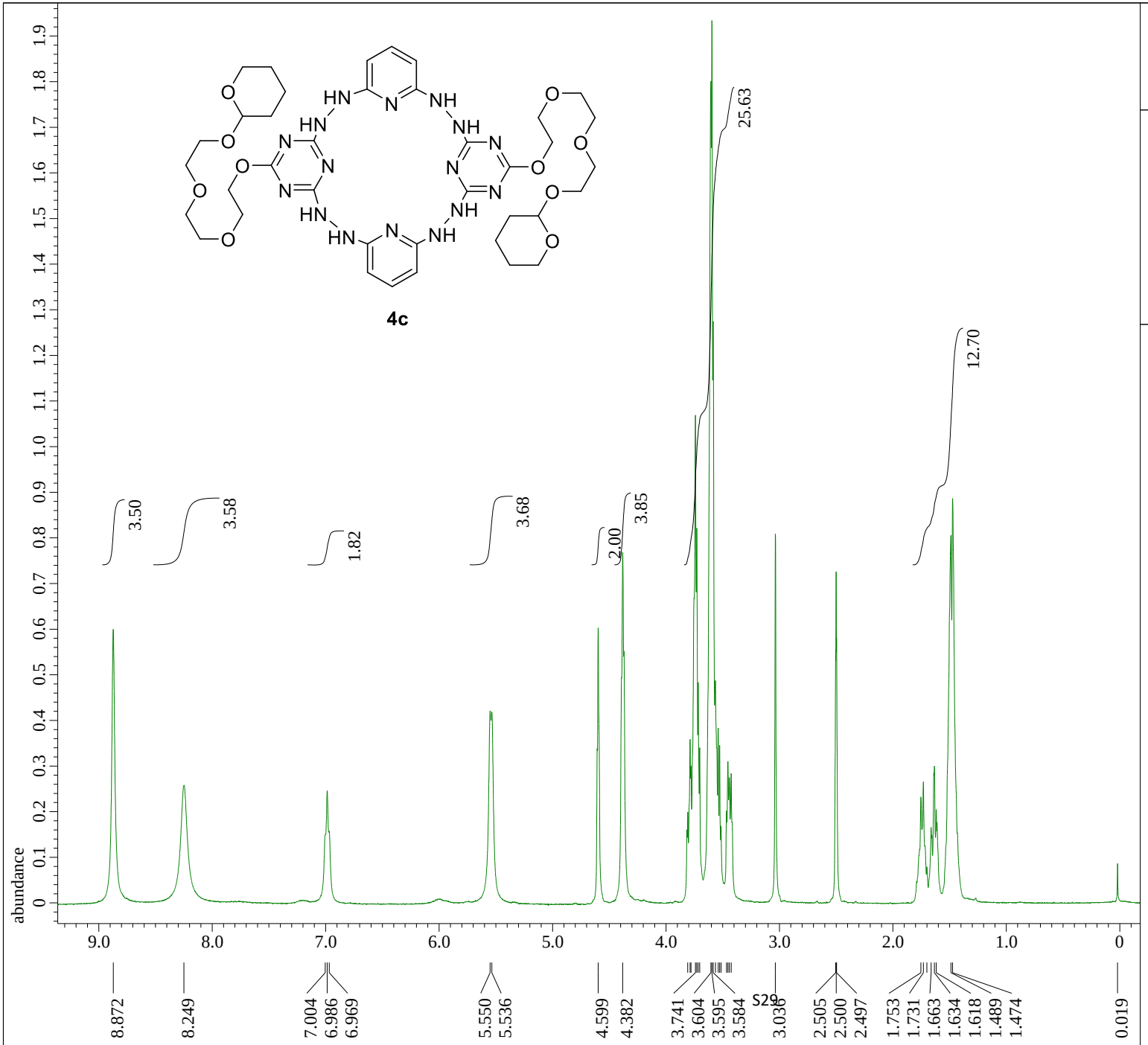

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = 4b-C-3.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = S#112045
Solvent           = DMSO-D6
Creation_Time     =  8-FEB-2017 03:25:04
Revision_Time     =  8-FEB-2017 03:35:35
Current_Time      = 16-FEB-2017 00:24:50

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 501
Total_Scans       = 501

Relaxation_Delay  = 2[s]
Recvr_Gain        = 54
Temp_Get          = 60[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 8.823[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 23.03[dB]
Irr_Atn_Noe       = 23.03[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
```



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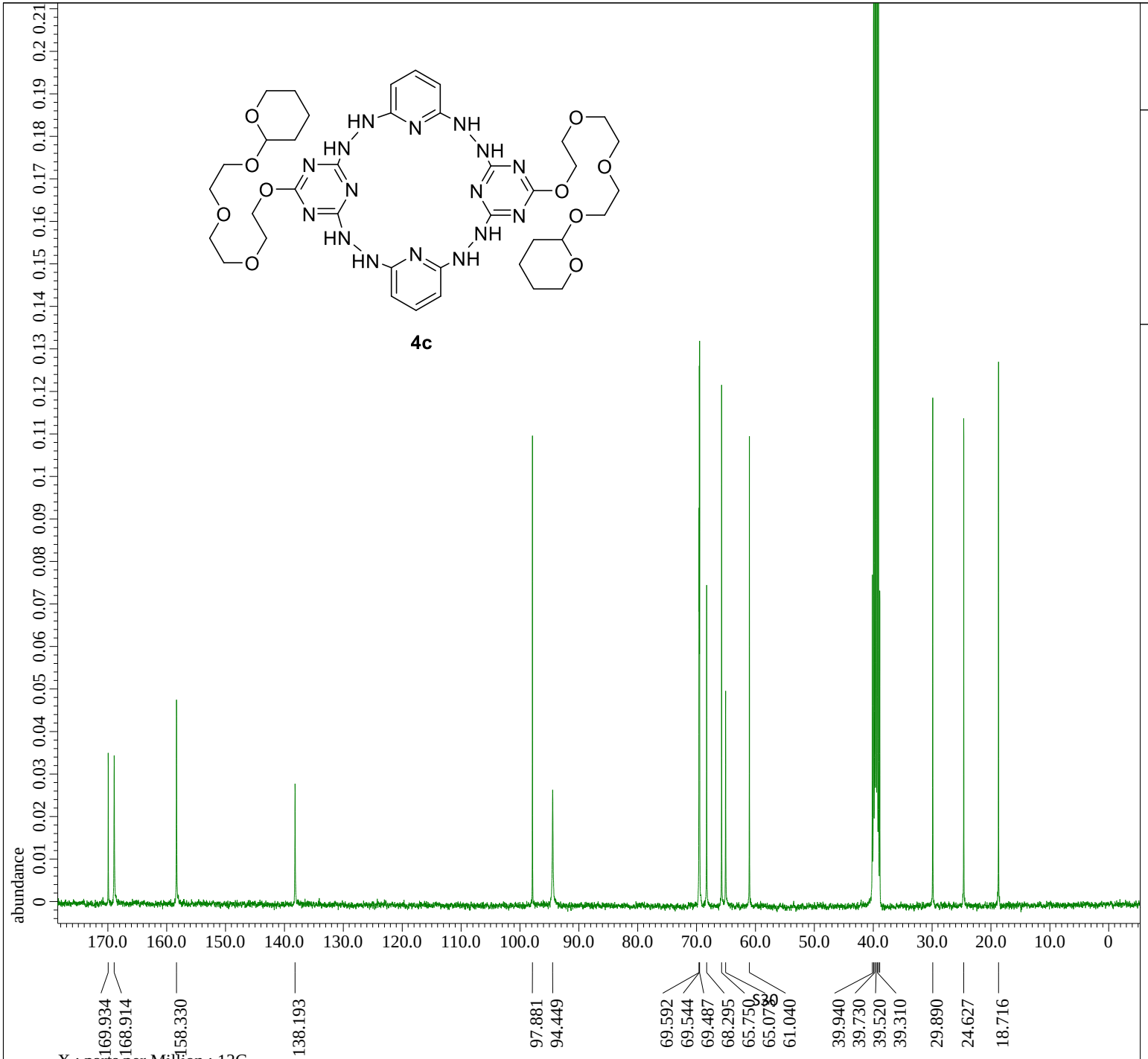
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 4c-H-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#831366
Solvent           = DMSO-D6
Creation_Time     = 16-MAR-2016 23:07:47
Revision_Time     = 10-FEB-2017 22:58:50
Current_Time      = 16-FEB-2017 00:25:28

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 44
Temp_Get          = 90[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```



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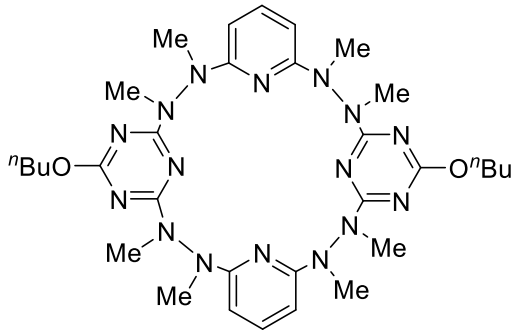
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

Filename	= 4c-C-3.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= S#848384
Solvent	= DMSO-D6
Creation_Time	= 17-MAR-2016 07:42:00
Revision_Time	= 17-MAR-2016 08:25:44
Current_Time	= 16-FEB-2017 00:26:56
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= JNM-ECX400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 9572
Total_Scans	= 9572
Relaxation_Delay	= 2[s]
Recvr_Gain	= 52
Temp_Get	= 90[dC]
X_90_Width	= 11[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 7.8[dB]
X_Pulse	= 3.66666667[us]
Irr_Atn_Dec	= 23.03[dB]
Irr_Atn_Noe	= 23.03[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]



5

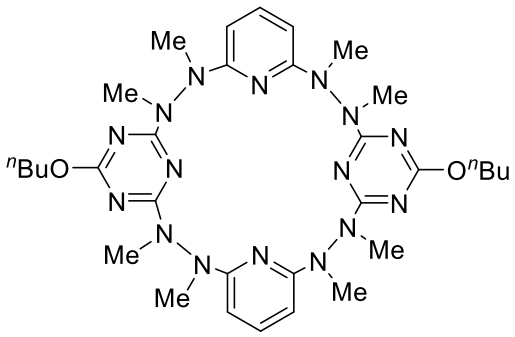

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
```

```
Filename          = 5-H-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#374595
Solvent           = CHLOROFORM-D
Creation_Time     = 26-MAR-2015 10:37:22
Revision_Time     = 16-FEB-2017 00:27:58
Current_Time      = 16-FEB-2017 00:28:02

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = JNM-ECX400

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 60
Temp_Get          = 20.4[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.4[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
```



5


```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0, 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
ppm
machinephase
```

Filename	= 5-C-9.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= S#90517
Solvent	= CHLOROFORM-D
Creation_Time	= 27-MAR-2015 08:35:24
Revision_Time	= 16-FEB-2017 00:28:54
Current_Time	= 16-FEB-2017 00:29:04
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= JNM-ECX400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 42
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= TRUE
Scans	= 6844
Total_Scans	= 6844
Relaxation_Delay	= 2[s]
Recvr_Gain	= 58
Temp_Get	= 19.9[dC]
X_90_Width	= 9.25[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 7.8[dB]
X_Pulse	= 3.08333333[us]
Irr_Atn_Dec	= 23.786[dB]
Irr_Atn_Noe	= 23.786[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]