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Synthesis and biological evaluation of novel phane-structured diazacrowns containing γ-piperidone and pyridine rings

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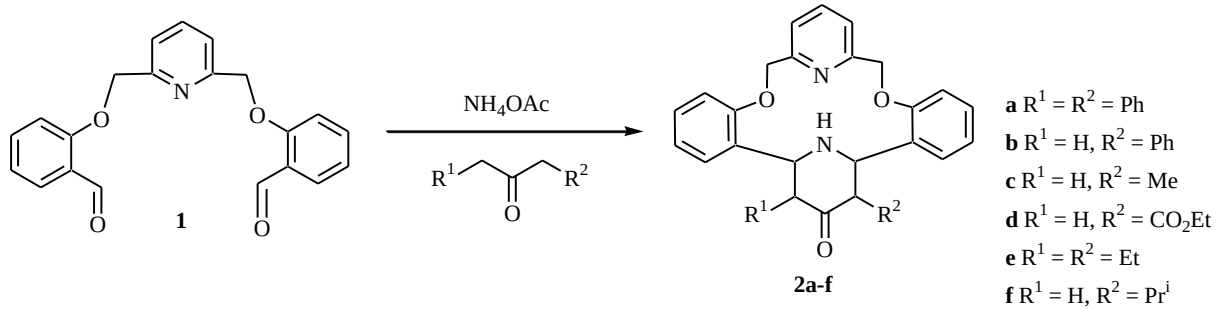
Contents

All new compounds gave satisfactory spectroscopic and analytical results. Their IR, MS, ^{1}H NMR and ^{13}C NMR spectra were consistent with the structures show in the Scheme S1.

2,6-Bis(2-formylphenoxymethyl)pyridine **1** was synthesized as reported [S1].

Synthesis of novel phane-structured diazacrowns 2a-f



Scheme S1. Synthesis of novel phane-structured diazacrowns **2a-f**

2,6-Bis(2-formylphenoxymethyl)pyridine **1** (0.25 g, 0.72 mmol) was added to a solution of ketone (1.1 mmol) and ammonium acetate (0.56 g, 7.2 mmol) in EtOH (20 ml) and AcOH (1 ml). The reaction mixture was stirred at 60 °C, and the reaction progress was mornitored by TLC. After 11–20 h, the solvent was removed under reduced pressure. Distilled water (30 ml) was added, and the mixture was neutralized with Na_2CO_3. Then, it was extracted with CH_2Cl_2 (3×30 ml), and the extracts were washed with a saturated solution of NaCl and dried over $MgSO_4$. Evaporation of solvent *in vacuo* gave a residue which was subjected to column chromatography with an elution n-hexane/ethyl acetate = 1:1 (650 ml). The proper fractions were evaporated, and the residue was recrystallized from CH_2Cl=2 to obtain pure white product **2a-f**.

*2^{3},2^{5}-Diphenyl-4,8-dioxa-6(2,6)-pyridina-2(2,6)-piperidina-1,3(1,2)-dibenzenacyclooctaphan-2^{4}-one (**2a**)*

Yield 45%, mp. 238-239°C. R_f = 0.52 (hexane:EtOAc = 1:1). ^{1}H-NMR (500 MHz, DMSO-D_6, TMS) δ_H ppm: 7.98 (1H, br. s, H-12), 7.59 (2H, br. s, H-11, H-13), 6.64 (2H, br. s, H-5, H-20), 6.90 (2H, br. s, H-6, H-19), 6.99 – 7.17 (14H, br. m, H-3, H-4, H-21, H-22, 2xPh), 5.36 (2H, br. s, H-1, H-24), 5.15 (2H, br. s, H-25, H-27), 4.97 (1H, br.s, NH-28), 4.46 (4H, br.s 2xH-9, 2xH-16). ^{13}C-NMR (125 MHz, DMSO-D_6) δ_C ppm: 157.22, 156.07, 137.65, 131.60, 129.30, 128.42, 127.35, 125.85, 121.19, 120.00, 111.80, 69.61, 69.00, 61.95. HRMS, *m/z:* 539.2336 $[M+H]^+$ and 561.2139 $[M+Na]^+$.Calc. for $C_{36}H_{31}N_2O_3^+$: 539.2329 and $C_{36}H_{30}N_2O_3Na^+$: 561.2149.

*2^{3}-Phenyl-4,8-dioxa-6(2,6)-pyridina-2(2,6)-piperidina-1,3(1,2)-dibenzenacyclooctaphan-2^{4}-one (**2b**)*

Yield 40%, mp. 211-212°C. R_f = 0.34 (hexane:EtOAc = 1:1). IR (KBr, *v/cm^{-1}*): 3263.56; 3057.17; 3028.24; 2920.23; 2872.01; 1710.86; 1593.20.^{1}H-NMR (500 MHz, $CDCl_3$, TMS) δ_H ppm: 7.78 (1H, t, J^3 8.0 Hz, H-12), 7.30 (2H, d, J^3 8.0 Hz, H-11, H-13), 6.66 (1H, t, J^3 7.5 Hz, H-20), 6.82 (1H, d, J^3 8.0 Hz, H-19), 6.95 – 6.99 (5H, m, 5xH_{Ph}), 7.05 (2H, t, J^3 7.5 Hz, H-4, H-21), 7.12 (2H, t, J^3 7.5 Hz, H-3, H-22), 7.28 – 7.34 (2H, m, H-5, H-6), 5.17 – 5.25 (4H, m, 2xH-9, 2xH-16), 4.34-4.37 (3H, br.m, H-24, H-25, H-27^{eq}), 3.37 (1H, t, J^3 13.0 Hz, H-1), 2.68 (1H, dd, J^3 14.0 Hz, J^5 3.0 Hz, H-27^{ax}), 1.26 (1H, br.s, NH-28). ^{13}C-NMR (125 MHz, $CDCl_3$) δ_C ppm: 157.47, 157.16, 156.33, 156.12, 137.29, 136.64, 129.31, 129.14, 128.60, 127.83, 126.46, 121.40, 121.03, 120.99, 120.95, 111.99, 70.35, 69.94, 62.26, 47.59, 40.27, 40.10, 39.94. HRMS, *m/z:* 463.2019 $[M+H]^+$ and 495.2275 $[M+MeOH+H]^+$.Calc. for $C_{30}H_{27}N_2O_3^+$: 463.2016 and $C_{31}H_{31}N_2O_4^+$: 495.2278.

*2^3-Methyl-4,8-dioxa-6(2,6)-pyridina-2(2,6)-piperidina-1,3(1,2)-dibenzenacyclooctaphan-2^4-one (**2c**)*

Yield 29%, mp. 217-218ºC. R_f = 0.61 (hexane:EtOAc = 1:1). IR (KBr, *v/cm^{-1}*): 3327.21, 3296.35, 3061.03, 2960.73, 2922.16, 2852.72, 1701.22, 1589.34.^{1}H-NMR (500 MHz, $CDCl_3$, TMS) δ_H ppm: 7.76 (1H, t, J^3 7.5 Hz, H-12), 7.31 (2H, d, J^3 8.0 Hz, H-11, H-13), 6.95 – 7.00 (4H, m, H-5, H-6, H-19, H-20), 7.25 – 7.29 (4H, m, H-4, H-21, H-3, H-22), 4.21 (1H, br.s, H-1), 3.70 (1H, br.s, H-24), 3.09 – 3.28 (2H, br.m, 2xH-27), 2.58 (1H, d, J^3 13.0 Hz, H-25), 1.72 (1H, br.s, NH-28), 5.16 (2H, s, 2xH-9), 5.19 – 5.25 (2H, br. m, 2xH-16), 0.83 (3H, d, J^3 6.0 Hz, H_{Me}). ^{13}C-NMR (125 MHz, $CDCl_3$) δ_C ppm: 213.12, 157.35, 156.26, 156.04, 137.24, 129.24, 129.08, 121.46, 120.95, 111.91, 69.94, 65.67, 49.26, 11.01. HRMS, *m/z:* 401.1863 $[M+H]^+$ and 433.2116 $[M+MeOH+H]^+$.Calc. for $C_{25}H_{25}N_2O_3^+$: 401.1860 and $C_{25}H_{29}N_2O_4^+$: 433.2122.

*Ethyl 2^4-oxo-4,8-dioxa-6(2,6)-pyridina-2(2,6)-piperidina-1,3(1,2)-dibenzenacyclooctaphane-2^3-carboxylate (**2d**)*

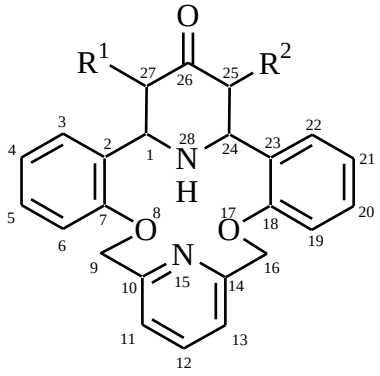
Yield 32%, mp. 198-199ºC. R_f = 0.45 (hexane:EtOAc = 2:3). ^{1}H-NMR (500 MHz, $CDCl_3$, TMS) δ_H ppm: 7.76 (1H, t, J^3 7.5 Hz, H-12), 7.30 (2H, d, J^3 7.0 Hz, H-11, H-13), 7.24 – 7.30 (4H, m, H-4, H-21, H-3, H-22), 6.88 – 6.96 (4H, m, H-5, H-6, H-19, H-20), 5.13 – 5.21 (4H, m, 2xH-9, 2xH-16), 4.40 (1H, br.s, H-1), 4.17 (1H, br.s, H-24), 3.98 - 4.02 (2H, br.m, -O-$\underline{CH_2}CH_3$), 3.21 (1H, br.s, H-27), 2.57 (1H, br.d, J^3 14.0 Hz, H-25), 1.03 (3H, t, J^3 7.0 Hz, -O-$CH_2\underline{CH_3}$). ^{13}C-NMR (125 MHz, $CDCl_3$) δ_C ppm: 204.93, 169.19, 157.61, 157.10, 156.10, 155.96, 137.28, 131.99, 130.75, 129.41, 129.27, 127.49, 121.37, 121.08, 120.98, 120.81, 111.95, 111.92, 89.90, 69.97, 69.86, 62.68, 60.54, 46.82, 13.98. HRMS, *m/z:* 459.1920 $[M+H]^+$ and 491.2183 $[M+MeOH+H]^+$.Calc. for $C_{27}H_{27}N_2O_5^+$: 459.1914 and $C_{28}H_{31}N_2O_6^+$: 491.2277.

*2^3,2^5-Diethyl-4,8-dioxa-6(2,6)-pyridina-2(2,6)-piperidina-1,3(1,2)-dibenzenacyclooctaphan-2^4-one (**2e**)*

Yield 26 %, mp. 219-220ºC. R_f = 0.38 (hexane:EtOAc = 1:1). ^{1}H-NMR (500 MHz, $CDCl_3$, TMS) δ_H ppm: 7.78 (1H, br.s, H-12), 7.32 (2H, d, J^3 8.0 Hz, H-11, H-13), 7.20 – 7.29 (4H, br. m, H-3, H-4, H-21, H-22), 6.90 – 6.98 (4H, br. m, H-5, H-6, H-19, H-20), 5.12 – 5.17 (4H, m, 2xH-9, 2xH-16), 3.68 (2H, br. s, H-1, H-24), 3.17 (2H, br. s, H-25, H-27), 1.40 – 1.60 (5H, br.m, NH-28, 2x-$\underline{CH_2}$-CH_3), 0,65 – 0.80 (6H, m, 2xH_{Me}). ^{13}C-NMR (125 MHz, $CDCl_3$) δ_C ppm: 211.80, 157.82, 156.26, 137.26, 132.45, 128.93, 121.09, 120.76, 114.40, 111.75, 70.25, 69.94, 57.54, 29.90, 19.47, 11.86. HRMS, *m/z:* 443.2331 $[M+H]^+$ .Calc. for $C_{28}H_{31}N_2O_3^+$: 443.2329.

*2^3-Isopropyl-4,8-dioxa-6(2,6)-pyridina-2(2,6)-piperidina-1,3(1,2)-dibenzenacyclooctaphan-2^4-one (**2f**)*

Yield 36%, mp. 240–241ºC. R_f = 0.45 (hexane:EtOAc = 1:1). ^{1}H-NMR (500 MHz, $CDCl_3$, TMS) δ_H ppm: 7.72 (1H, t, J^3 8.0 Hz, H-12), 7.34 (2H, br.s, H-11, H-13), 6.91 – 6.97 (4H, m, H-5, H-6, H-19, H-20), 7.18 – 7.43 (4H, m, H-4, H-21, H-3, H-22), 5.09 – 5.22 (4H, br.m, 2xH-9, 2xH-16), 4.21 (2H, br.s, H-1, H-24), 3.03 – 3.21 (2H, br.m, 2xH-27), 2.50 (1H, br.d, J^3 13.0 Hz, H-25), 1.75 (1H, br.s, NH-28), 0.79 (3H, d., J^3 7.5 Hz, H_{Me}), 0.98 (3H, d, J^3 6.5 Hz, H_{Me}), 1.75 (1H, br. s, $H_{\text{-CH-isopropyl}}$). ^{13}C-NMR (125 MHz, CDC_{l3}) δ_C ppm: 210.19, 156.40, 156.06, 137.20, 129.09, 121.44, 120.93, 111.88, 69.82, 67.43, 58.99, 48.68, 42.11, 29.70, 26.58, 20.66, 17.46. HRMS, m/z: 429.2173 $[M+H]^+$, 451.2119 $[M+Na]^+$ and 461.2438 $[M+MeOH+H]^+$. Calc. for $C_{27}H_{29}N_2O_3^+$: 429.2173, $C_{27}H_{28}N_2NaO_3^+$: 451.1992 and $C_{28}H_{33}N_2O_4^+$: 461.2435.



2a-f

2a: $R^1 = R^2$ = Ph
2b: R^1 = H, R^2 = Ph
2c: R^1 = H, R^2 = Me
2d: R^1 = H, R^2 = COOEt;
2e: $R^1 = R^2$ = Et
2f: R^1 = H, R^2 = Pr^i

Biological evaluation

Cytotoxicity assay. Vero cell line and five human cancer cell lines were obtained from the American Type Culture Collection (Manassas, VA) ATCC as RD (human rhabdomyosarcoma), Hep-G2 (human hepatocellular carcinoma), MCF7 (human breast adenocarcinoma), Lu-1 (human lung adenocarcinoma). The MTT is based on the protocol described by Skehan & etc (1990) [S2] and Likhiwitayawuid & etc (1993) [S3]. This method has worldwide application and is recommended by National Cancer Institute (NCI) and College of Medicine, University of Illinois at Chicago for routine drug screening.

Antibacterial activity assay. A high-throughput 96-well microplate bioassay procedure was used according to the method of Vanden Berghe and Vlietlinck [S4] and McKane and Kandel [S5]. A macrobroth dilution method was used to determine the MIC values. The antibacterial stock solution was made at the final concentration of 50 μg/ml. All plates were incubated aerobically at 37°C for 24 h for bacteria and at 30°C for 48 h for fungi.

Table S1. *Antibacterial activity of compounds **2a-c,e,f.***

Comp.	**Conc. μg ml^{-1}**	**Minimum Inhibitory Concentration (MIC: μg ml^{-1})**								**Conclusion**
		Gram-negative bacteria		***Gram-positive bacteria***		***Filamentous fungi***		***Yeast fungi***		
		E. coli	*P. aeruginosa*	*B. subtilis*	*S. aureus*	*A. niger*	*F. oxysporum*	*S. cerevisiae*	*C. albicans*	
2a	**200**	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	Negative
2b	**200**	(-)	(-)	**100**	(-)	**100**	(-)	(-)	(-)	Positive with 01 Gr-(+) bacteria & 01 fungi lines
2c	**200**	(-)	(-)	**100**	(-)	**100**	(-)	(-)	(-)	
2e	**200**	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	Negative
2f	**200**	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	Negative

X-ray diffraction experimental data

Experimental details

Crystal data	
Chemical formula ***2a • 2CHCl₃***	$C_{36}H_{30}N_2O_3 \cdot 2(CHCl_3)$
M_r	777.36
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	120
a, b, c (Å)	10.9906 (16), 19.149 (3), 34.090 (5)
β (°)	96.159 (2)
V (Å^3)	7133.1 (18)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.52
Crystal size (mm)	0.30 × 0.20 × 0.20
Data collection	
Diffractometer	Bruker *APEX*-II CCD
Absorption correction	Multi-scan *SADABS*
T_{min}, T_{max}	0.848, 0.888
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	46553, 10332, 8144
R_{int}	0.064
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.606
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.107, 0.253, 1.06
No. of reflections	10332
No. of parameters	892
No. of restraints	12
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
	$w = 1/[\sigma^2(F_o^2) + (0.01P)^2 + 13P]$ where $P = (F_o^2 + 2F_c^2)/3$
$\Delta\rangle_{max}$, $\Delta\rangle_{min}$ (e Å^{-3})	1.46, -2.13

Table S2. *Hydrogen bonds for* ***2a • 2CHCl₃*** *[Å and °].*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N28—H28···O8	0.81 (9)	2.63 (8)	3.017 (9)	111 (7)
N28—H28···O16	0.81 (9)	2.51 (9)	3.053 (9)	126 (7)
N28—H28···N27	0.81 (9)	2.66 (9)	3.452 (10)	169 (8)
N28*A*—H28*A*···O8*A*	0.78 (9)	2.51 (9)	2.995 (9)	121 (7)
N28*A*—H28*A*···O16*A*	0.78 (9)	2.58 (8)	3.023 (9)	118 (7)
N28*A*—H28*A*···N27*A*	0.78 (9)	2.68 (9)	3.455 (10)	174 (8)
C41—H41···N28	1.00	2.22	3.101 (10)	146
C42—H42···O25	1.00	2.33	3.151 (10)	139
C43—H43···N28*A*	1.00	2.17	3.088 (10)	151
C44—H44*A*···O25*A*	1.00	2.09	3.060 (7)	162

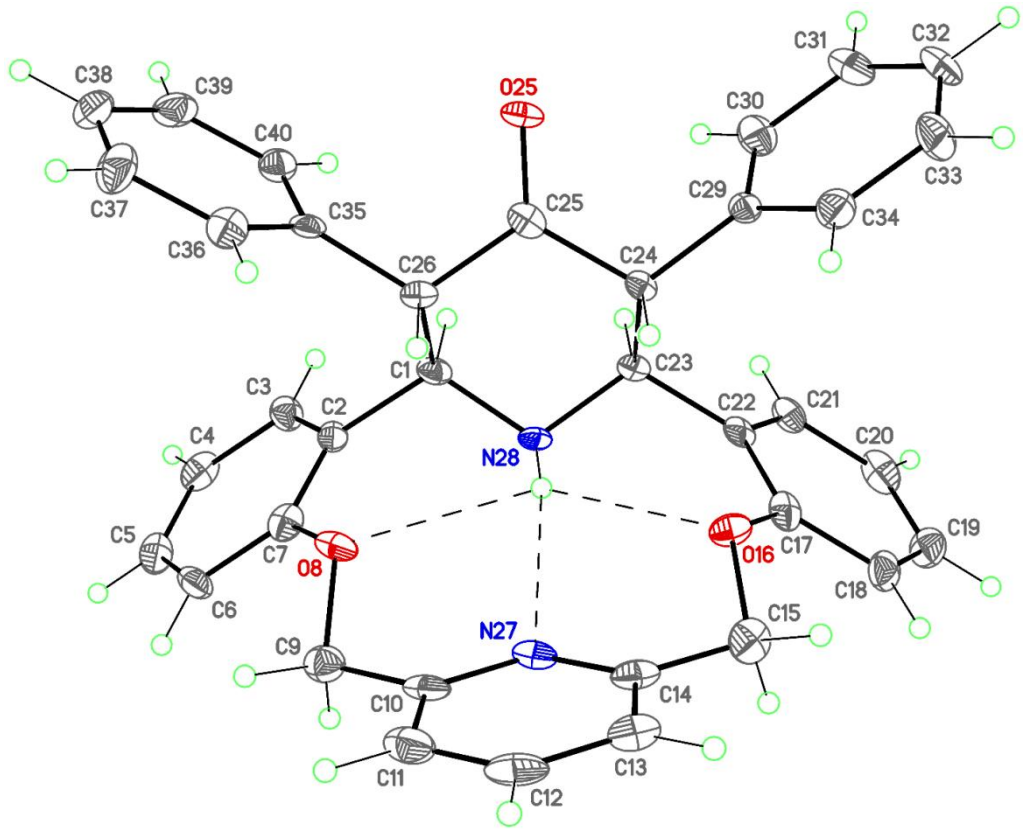


Figure S1 Molecular structure of **2a** (one of the two crystallographically independent molecules is presented only). The intramolecular N─H···O and N─H···N hydrogen bonds are depicted by dashed lines.

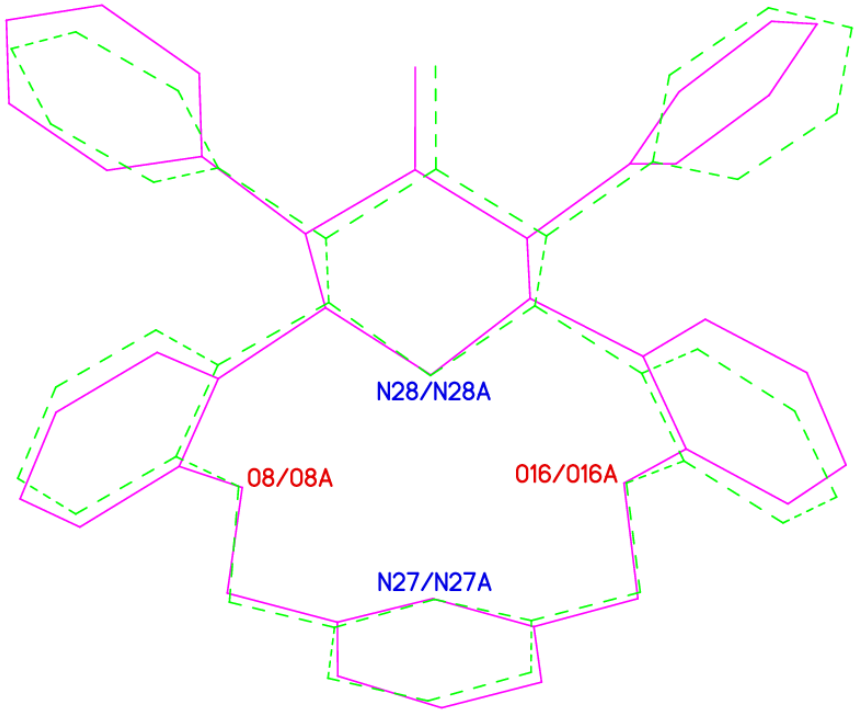


Figure S2 Superposition of the two crystallographically independent molecules of **2a**. The atoms those were imposed to each other are labelled.

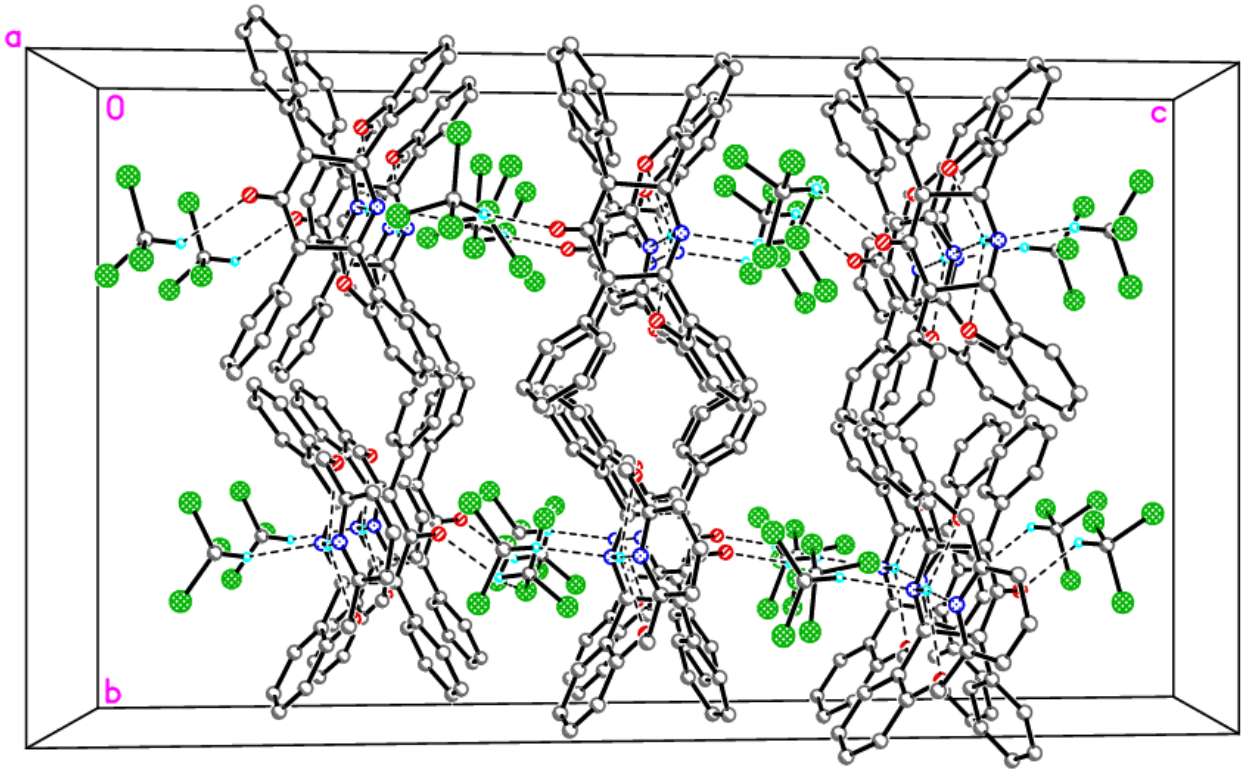


Figure S3. Crystal packing of **2a • 2CHCl$_3$.** The intramolecular N─H···O and N─H···N and intermolecular C─H···O and C─H···N hydrogen bonds are depicted by dashed lines.

Full crystallographic data for 2a

Crystal data 2a • 2CHCl₃

$C_{36}H_{30}N_2O_3 \cdot 2(CHCl_3)$	$F(000) = 3200$
$M_r = 777.36$	$D_x = 1.448$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 10.9906$ (16) Å	Cell parameters from 9870 reflections
$b = 19.149$ (3) Å	$\theta = 2.2–26.4°$
$c = 34.090$ (5) Å	$\mu = 0.52$ mm^{-1}
$\beta = 96.159$ (2)°	$T = 120$ K
$V = 7133.1$ (18) Å^3	Prism, yellow
$Z = 8$	0.30 × 0.20 × 0.20 mm

Data collection

Bruker APEX-II CCD diffractometer	8144 reflections with $I > 2\sigma(I)$
Radiation source: normal-focus sealed tube	$R_{int} = 0.064$
ϕ and ω scans	$\theta_{max} = 25.5°$, $\theta_{min} = 1.2°$
Absorption correction: multi-scan *SADABS*	$h = -12 \rightarrow 12$
$T_{min} = 0.848$, $T_{max} = 0.888$	$k = -23 \rightarrow 23$
46553 measured reflections	$l = -41 \rightarrow 41$
10332 independent reflections	

Refinement

Refinement on F^2	Primary atom site location: difference Fourier map
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.107$	Hydrogen site location: mixed
$wR(F^2) = 0.253$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.06$	$w = 1/[\sigma^2(F_o^2) + (0.01P)^2 + 13P]$ where $P = (F_o^2 + 2F_c^2)/3$
10332 reflections	$(\Delta/\sigma)_{max} = 0.001$
892 parameters	$\Delta\rangle_{max} = 1.46$ e Å^{-3}
12 restraints	$\Delta\rangle_{min} = -2.13$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2) for ***(2a)***

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.9918 (8)	0.1587 (4)	0.2678 (2)	0.0203 (18)	
H1	1.0804	0.1682	0.2760	0.024*	
C2	0.9605 (8)	0.0945 (4)	0.2906 (2)	0.0188 (18)	
C3	1.0512 (8)	0.0639 (4)	0.3167 (2)	0.0253 (19)	
H3	1.1317	0.0827	0.3194	0.030*	
C4	1.0252 (9)	0.0062 (4)	0.3389 (2)	0.028 (2)	
H4	1.0875	-0.0145	0.3566	0.033*	
C5	0.9100 (9)	-0.0207 (4)	0.3351 (2)	0.030 (2)	
H5	0.8923	-0.0598	0.3507	0.036*	
C6	0.8194 (9)	0.0076 (4)	0.3094 (2)	0.027 (2)	
H6	0.7395	-0.0120	0.3069	0.032*	
C7	0.8450 (8)	0.0653 (4)	0.2869 (2)	0.0241 (19)	
O8	0.7608 (5)	0.0965 (3)	0.26090 (15)	0.0241 (13)	
C9	0.6362 (8)	0.0888 (4)	0.2673 (2)	0.027 (2)	
H9A	0.6272	0.0908	0.2958	0.033*	
H9B	0.6045	0.0432	0.2569	0.033*	
C10	0.5669 (7)	0.1471 (4)	0.2461 (2)	0.0244 (19)	
C11	0.4607 (8)	0.1360 (5)	0.2219 (3)	0.033 (2)	
H11	0.4294	0.0900	0.2177	0.039*	
C12	0.4001 (8)	0.1920 (5)	0.2037 (3)	0.032 (2)	
H12	0.3266	0.1854	0.1867	0.039*	
C13	0.4475 (8)	0.2571 (5)	0.2105 (2)	0.027 (2)	
H13	0.4074	0.2968	0.1984	0.032*	
C14	0.5562 (8)	0.2647 (5)	0.2355 (2)	0.026 (2)	
C15	0.6100 (8)	0.3341 (4)	0.2458 (2)	0.027 (2)	
H15A	0.5767	0.3693	0.2262	0.033*	
H15B	0.5894	0.3486	0.2721	0.033*	
O16	0.7391 (5)	0.3291 (3)	0.24581 (15)	0.0231 (13)	
C17	0.8127 (8)	0.3705 (4)	0.2706 (2)	0.0225 (19)	
C18	0.7710 (8)	0.4325 (4)	0.2867 (2)	0.026 (2)	
H18	0.6887	0.4473	0.2803	0.031*	
C19	0.8497 (9)	0.4715 (4)	0.3118 (2)	0.030 (2)	
H19	0.8215	0.5133	0.3229	0.035*	
C20	0.9683 (9)	0.4509 (4)	0.3209 (2)	0.032 (2)	
H20	1.0223	0.4781	0.3384	0.038*	
C21	1.0097 (8)	0.3900 (4)	0.3046 (2)	0.0247 (19)	
H21	1.0923	0.3759	0.3110	0.030*	

C22	0.9321 (7)	0.3495 (4)	0.2791 (2)	0.0187 (18)	
C23	0.9801 (7)	0.2829 (4)	0.2629 (2)	0.0149 (16)	
H23	1.0690	0.2814	0.2729	0.018*	
C24	0.9753 (7)	0.2817 (4)	0.2171 (2)	0.0155 (16)	
H24	0.8871	0.2817	0.2062	0.019*	
C25	1.0301 (7)	0.2138 (4)	0.2048 (2)	0.0185 (17)	
O25	1.1093 (5)	0.2117 (3)	0.18263 (15)	0.0238 (13)	
C26	0.9804 (8)	0.1486 (4)	0.2222 (2)	0.0187 (17)	
H26	0.8912	0.1460	0.2127	0.022*	
N27	0.6146 (6)	0.2104 (3)	0.25336 (18)	0.0218 (15)	
N28	0.9268 (6)	0.2207 (3)	0.27856 (18)	0.0167 (14)	
H28	0.855 (8)	0.222 (4)	0.270 (3)	0.020*	
C29	1.0331 (7)	0.3451 (4)	0.2010 (2)	0.0182 (18)	
C30	1.1543 (8)	0.3602 (4)	0.2109 (2)	0.029 (2)	
H30	1.2032	0.3296	0.2279	0.035*	
C31	1.2070 (9)	0.4193 (5)	0.1964 (3)	0.035 (2)	
H31	1.2911	0.4292	0.2037	0.042*	
C32	1.1365 (9)	0.4637 (5)	0.1715 (3)	0.035 (2)	
H32	1.1721	0.5042	0.1614	0.042*	
C33	1.0160 (9)	0.4494 (5)	0.1612 (3)	0.035 (2)	
H33	0.9675	0.4802	0.1442	0.042*	
C34	0.9645 (8)	0.3904 (4)	0.1755 (2)	0.0262 (19)	
H34	0.8806	0.3804	0.1678	0.031*	
C35	1.0389 (7)	0.0824 (4)	0.2095 (2)	0.0190 (18)	
C36	0.9733 (8)	0.0370 (4)	0.1842 (2)	0.0256 (19)	
H36	0.8927	0.0493	0.1736	0.031*	
C37	1.0215 (9)	-0.0263 (5)	0.1738 (3)	0.037 (2)	
H37	0.9737	-0.0570	0.1565	0.044*	
C38	1.1376 (9)	-0.0445 (5)	0.1883 (3)	0.034 (2)	
H38	1.1712	-0.0880	0.1814	0.041*	
C39	1.2049 (8)	0.0008 (5)	0.2129 (3)	0.032 (2)	
H39	1.2862	-0.0112	0.2229	0.038*	
C40	1.1562 (8)	0.0637 (4)	0.2235 (2)	0.027 (2)	
H40	1.2044	0.0944	0.2406	0.032*	
C1A	0.2222 (7)	0.2006 (4)	0.5160 (2)	0.0151 (16)	
H1A	0.3103	0.2030	0.5269	0.018*	
C2A	0.1718 (8)	0.1341 (4)	0.5329 (2)	0.0191 (18)	
C3A	0.2496 (8)	0.0921 (4)	0.5569 (2)	0.0250 (19)	
H3A	0.3324	0.1059	0.5633	0.030*	
C4A	0.2087 (9)	0.0295 (4)	0.5720 (2)	0.030 (2)	
H4A	0.2631	0.0003	0.5881	0.036*	

C5A	0.0891 (8)	0.0113 (4)	0.5629 (2)	0.027 (2)	
H5A	0.0601	-0.0310	0.5732	0.032*	
C6A	0.0101 (8)	0.0524 (4)	0.5395 (2)	0.0252 (19)	
H6A	-0.0731	0.0388	0.5337	0.030*	
C7A	0.0514 (7)	0.1140 (4)	0.5242 (2)	0.0183 (18)	
O8A	-0.0214 (5)	0.1571 (3)	0.49969 (16)	0.0230 (13)	
C9A	-0.1485 (7)	0.1506 (4)	0.4986 (2)	0.0250 (19)	
H9C	-0.1706	0.1360	0.5248	0.030*	
H9D	-0.1788	0.1149	0.4790	0.030*	
C10A	-0.2052 (8)	0.2201 (4)	0.4875 (2)	0.0232 (19)	
C11A	-0.3156 (8)	0.2259 (5)	0.4634 (2)	0.029 (2)	
H11A	-0.3547	0.1857	0.4516	0.035*	
C12A	-0.3664 (8)	0.2908 (5)	0.4570 (2)	0.029 (2)	
H12A	-0.4428	0.2961	0.4414	0.035*	
C13A	-0.3052 (8)	0.3479 (5)	0.4735 (2)	0.030 (2)	
H13A	-0.3378	0.3936	0.4692	0.036*	
C14A	-0.1950 (8)	0.3378 (4)	0.4966 (2)	0.026 (2)	
C15A	-0.1257 (8)	0.3969 (4)	0.5158 (2)	0.029 (2)	
H15C	-0.1566	0.4417	0.5042	0.034*	
H15D	-0.1340	0.3975	0.5445	0.034*	
O16A	-0.0015 (5)	0.3870 (3)	0.50929 (16)	0.0244 (13)	
C17A	0.0863 (7)	0.4195 (4)	0.5341 (2)	0.0185 (18)	
C18A	0.0620 (9)	0.4786 (4)	0.5558 (2)	0.027 (2)	
H18A	-0.0182	0.4978	0.5537	0.033*	
C19A	0.1537 (9)	0.5086 (4)	0.5798 (2)	0.028 (2)	
H19A	0.1369	0.5488	0.5947	0.034*	
C20A	0.2695 (9)	0.4822 (4)	0.5830 (2)	0.026 (2)	
H20A	0.3335	0.5039	0.5996	0.032*	
C21A	0.2923 (8)	0.4233 (4)	0.5615 (2)	0.0221 (18)	
H21A	0.3727	0.4045	0.5638	0.027*	
C22A	0.2013 (8)	0.3907 (4)	0.5367 (2)	0.0203 (18)	
C23A	0.2298 (7)	0.3247 (4)	0.5154 (2)	0.0153 (16)	
H23A	0.3190	0.3156	0.5227	0.018*	
C24A	0.2123 (7)	0.3297 (4)	0.4693 (2)	0.0152 (16)	
H24A	0.1224	0.3298	0.4606	0.018*	
C25A	0.2656 (7)	0.2628 (4)	0.4543 (2)	0.0149 (16)	
O25A	0.3419 (5)	0.2648 (3)	0.43090 (15)	0.0228 (13)	
C26A	0.2215 (7)	0.1949 (4)	0.4702 (2)	0.0161 (16)	
H26A	0.1346	0.1883	0.4588	0.019*	
N27A	-0.1464 (6)	0.2745 (4)	0.50404 (18)	0.0234 (16)	
N28A	0.1661 (6)	0.2638 (3)	0.52883 (19)	0.0168 (14)	

H28A	0.096 (8)	0.265 (4)	0.521 (3)	0.020*	
C29A	0.2670 (7)	0.3934 (4)	0.4524 (2)	0.0169 (17)	
C30A	0.3794 (8)	0.4189 (4)	0.4668 (2)	0.0244 (19)	
H30A	0.4238	0.3966	0.4888	0.029*	
C31A	0.4297 (8)	0.4765 (4)	0.4500 (2)	0.028 (2)	
H31A	0.5082	0.4930	0.4602	0.033*	
C32A	0.3651 (9)	0.5098 (4)	0.4184 (3)	0.030 (2)	
H32A	0.3985	0.5497	0.4069	0.036*	
C33A	0.2529 (8)	0.4850 (4)	0.4036 (2)	0.029 (2)	
H33A	0.2081	0.5076	0.3818	0.035*	
C34A	0.2048 (8)	0.4270 (4)	0.4204 (2)	0.0237 (19)	
H34A	0.1271	0.4098	0.4097	0.028*	
C35A	0.2925 (7)	0.1323 (4)	0.4589 (2)	0.0180 (17)	
C36A	0.2347 (8)	0.0789 (4)	0.4370 (2)	0.0238 (19)	
H36A	0.1505	0.0830	0.4275	0.029*	
C37A	0.2990 (9)	0.0194 (4)	0.4288 (3)	0.032 (2)	
H37A	0.2581	-0.0176	0.4143	0.039*	
C38A	0.4223 (9)	0.0136 (4)	0.4416 (3)	0.031 (2)	
H38A	0.4665	-0.0271	0.4360	0.037*	
C39A	0.4798 (8)	0.0675 (5)	0.4627 (3)	0.030 (2)	
H39A	0.5646	0.0644	0.4715	0.036*	
C40A	0.4143 (8)	0.1266 (4)	0.4711 (2)	0.025 (2)	
H40A	0.4551	0.1637	0.4857	0.030*	
Cl1	0.8828 (3)	0.30590 (12)	0.39591 (8)	0.0472 (7)	
Cl2	0.8733 (3)	0.15726 (12)	0.38747 (7)	0.0475 (7)	
Cl3	1.0896 (2)	0.23260 (15)	0.37201 (7)	0.0427 (6)	
C41	0.9313 (8)	0.2347 (4)	0.3693 (2)	0.027 (2)	
H41	0.8978	0.2401	0.3410	0.032*	
Cl4	1.0935 (3)	0.32645 (16)	0.06903 (8)	0.0545 (8)	
Cl5	1.3319 (3)	0.3091 (2)	0.10863 (8)	0.0696 (10)	
Cl6	1.1806 (4)	0.18989 (15)	0.09047 (11)	0.0851 (13)	
C42	1.1846 (10)	0.2774 (5)	0.1040 (3)	0.039 (2)	
H42	1.1511	0.2820	0.1300	0.046*	
Cl7	0.3574 (2)	0.27545 (14)	0.62016 (7)	0.0406 (6)	
Cl8	0.1505 (3)	0.34511 (13)	0.64674 (8)	0.0551 (8)	
Cl9	0.1504 (2)	0.19647 (11)	0.63969 (7)	0.0373 (6)	
C43	0.1991 (9)	0.2747 (4)	0.6197 (2)	0.029 (2)	
H43	0.1612	0.2787	0.5918	0.035*	
Cl10	0.5725 (3)	0.2698 (2)	0.35177 (17)	0.0649 (9)	0.7
Cl11	0.3402 (4)	0.2480 (2)	0.30635 (12)	0.0649 (9)	0.7
Cl12	0.4484 (5)	0.13767 (16)	0.35572 (14)	0.0649 (9)	0.7

Cl1A	0.5507 (7)	0.2875 (4)	0.3479 (3)	0.064 (2)	0.3
Cl1B	0.3423 (9)	0.2196 (5)	0.3056 (2)	0.064 (2)	0.3
Cl1C	0.4921 (9)	0.1463 (3)	0.3669 (2)	0.064 (2)	0.3
C44	0.4300 (3)	0.22818 (19)	0.35125 (11)	0.056 (3)	
H44A	0.3875	0.2459	0.3738	0.067*	0.7
H44B	0.3767	0.2457	0.3711	0.067*	0.3

Atomic displacement parameters (Å^2) for (2a)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.022 (5)	0.024 (4)	0.016 (4)	0.001 (3)	0.005 (3)	0.005 (3)
C2	0.028 (5)	0.016 (4)	0.014 (4)	-0.002 (3)	0.006 (4)	0.001 (3)
C3	0.033 (6)	0.023 (4)	0.020 (4)	0.000 (4)	0.002 (4)	0.001 (3)
C4	0.038 (6)	0.028 (4)	0.017 (4)	0.013 (4)	0.001 (4)	0.005 (4)
C5	0.048 (7)	0.020 (4)	0.022 (4)	0.003 (4)	0.008 (4)	0.003 (4)
C6	0.036 (6)	0.023 (4)	0.024 (4)	-0.011 (4)	0.013 (4)	-0.002 (4)
C7	0.036 (6)	0.026 (4)	0.011 (4)	0.005 (4)	0.001 (4)	-0.004 (3)
O8	0.019 (3)	0.033 (3)	0.022 (3)	-0.008 (3)	0.007 (3)	0.001 (3)
C9	0.027 (6)	0.030 (5)	0.026 (4)	-0.003 (4)	0.005 (4)	-0.004 (4)
C10	0.014 (5)	0.039 (5)	0.022 (4)	-0.007 (4)	0.009 (4)	-0.006 (4)
C11	0.021 (6)	0.042 (5)	0.036 (5)	-0.009 (4)	0.011 (4)	-0.009 (4)
C12	0.009 (5)	0.059 (6)	0.028 (5)	0.000 (4)	0.002 (4)	-0.008 (5)
C13	0.014 (5)	0.041 (5)	0.025 (4)	0.005 (4)	0.002 (4)	-0.003 (4)
C14	0.017 (5)	0.041 (5)	0.023 (4)	0.003 (4)	0.013 (4)	0.001 (4)
C15	0.025 (6)	0.035 (5)	0.023 (4)	0.006 (4)	0.000 (4)	0.008 (4)
O16	0.019 (3)	0.031 (3)	0.019 (3)	0.002 (2)	-0.001 (2)	-0.001 (2)
C17	0.035 (6)	0.020 (4)	0.012 (4)	-0.001 (4)	0.003 (4)	0.004 (3)
C18	0.032 (6)	0.022 (4)	0.024 (4)	0.003 (4)	0.004 (4)	0.006 (4)
C19	0.035 (6)	0.027 (4)	0.028 (5)	0.001 (4)	0.008 (4)	-0.004 (4)
C20	0.047 (7)	0.027 (5)	0.020 (4)	-0.015 (4)	0.002 (4)	-0.005 (4)
C21	0.029 (5)	0.024 (4)	0.021 (4)	-0.003 (4)	0.005 (4)	0.005 (4)
C22	0.023 (5)	0.020 (4)	0.014 (4)	0.001 (3)	0.002 (3)	0.009 (3)
C23	0.006 (4)	0.023 (4)	0.015 (4)	-0.003 (3)	-0.003 (3)	0.003 (3)
C24	0.017 (5)	0.017 (4)	0.011 (4)	-0.001 (3)	-0.002 (3)	0.005 (3)
C25	0.013 (5)	0.027 (4)	0.013 (4)	-0.001 (3)	-0.007 (3)	0.007 (3)
O25	0.020 (3)	0.033 (3)	0.020 (3)	-0.001 (2)	0.009 (3)	0.004 (2)
C26	0.020 (5)	0.023 (4)	0.014 (4)	0.001 (3)	0.002 (3)	0.003 (3)
N27	0.016 (4)	0.031 (4)	0.019 (3)	-0.004 (3)	0.005 (3)	0.000 (3)
N28	0.014 (4)	0.022 (3)	0.014 (3)	0.002 (3)	0.001 (3)	0.003 (3)
C29	0.022 (5)	0.017 (4)	0.016 (4)	0.000 (3)	0.006 (3)	-0.002 (3)
C30	0.033 (6)	0.032 (5)	0.022 (4)	0.000 (4)	0.002 (4)	0.007 (4)
C31	0.040 (6)	0.037 (5)	0.030 (5)	-0.012 (4)	0.011 (4)	0.004 (4)

C32	0.042 (7)	0.027 (5)	0.038 (5)	-0.010 (4)	0.015 (5)	0.009 (4)
C33	0.042 (7)	0.036 (5)	0.027 (5)	0.003 (4)	0.003 (4)	0.014 (4)
C34	0.023 (5)	0.027 (4)	0.028 (5)	0.000 (4)	0.003 (4)	-0.001 (4)
C35	0.018 (5)	0.024 (4)	0.018 (4)	-0.001 (3)	0.010 (3)	0.006 (3)
C36	0.025 (5)	0.029 (4)	0.023 (4)	-0.001 (4)	0.001 (4)	0.001 (4)
C37	0.048 (7)	0.031 (5)	0.030 (5)	0.002 (4)	0.004 (5)	-0.010 (4)
C38	0.042 (7)	0.030 (5)	0.034 (5)	0.008 (4)	0.016 (5)	-0.002 (4)
C39	0.021 (5)	0.038 (5)	0.037 (5)	0.010 (4)	0.010 (4)	0.006 (4)
C40	0.031 (6)	0.029 (4)	0.021 (4)	-0.003 (4)	0.007 (4)	-0.002 (4)
C1A	0.011 (4)	0.020 (4)	0.015 (4)	-0.004 (3)	0.001 (3)	-0.002 (3)
C2A	0.030 (5)	0.015 (4)	0.013 (4)	-0.002 (3)	0.008 (4)	-0.006 (3)
C3A	0.029 (5)	0.028 (4)	0.018 (4)	0.003 (4)	0.000 (4)	-0.004 (4)
C4A	0.044 (7)	0.026 (4)	0.021 (4)	0.003 (4)	0.006 (4)	0.006 (4)
C5A	0.040 (6)	0.018 (4)	0.024 (4)	-0.003 (4)	0.012 (4)	0.007 (4)
C6A	0.026 (5)	0.024 (4)	0.027 (4)	-0.007 (4)	0.010 (4)	-0.003 (4)
C7A	0.021 (5)	0.019 (4)	0.016 (4)	-0.003 (3)	0.006 (3)	-0.006 (3)
O8A	0.022 (4)	0.025 (3)	0.022 (3)	-0.004 (2)	0.002 (2)	0.006 (2)
C9A	0.018 (5)	0.031 (5)	0.026 (4)	-0.008 (4)	0.005 (4)	-0.004 (4)
C10A	0.017 (5)	0.036 (5)	0.018 (4)	-0.007 (4)	0.005 (4)	0.000 (4)
C11A	0.018 (5)	0.044 (5)	0.028 (5)	-0.010 (4)	0.010 (4)	-0.007 (4)
C12A	0.011 (5)	0.051 (6)	0.026 (4)	-0.007 (4)	0.003 (4)	0.005 (4)
C13A	0.024 (6)	0.039 (5)	0.028 (5)	0.009 (4)	0.010 (4)	0.010 (4)
C14A	0.025 (5)	0.034 (5)	0.022 (4)	0.004 (4)	0.011 (4)	0.002 (4)
C15A	0.031 (6)	0.030 (5)	0.026 (4)	0.006 (4)	0.006 (4)	0.002 (4)
O16A	0.020 (4)	0.030 (3)	0.024 (3)	0.006 (3)	0.008 (3)	-0.003 (2)
C17A	0.022 (5)	0.016 (4)	0.017 (4)	-0.007 (3)	0.002 (4)	0.003 (3)
C18A	0.033 (6)	0.025 (4)	0.025 (4)	0.004 (4)	0.010 (4)	0.001 (4)
C19A	0.042 (6)	0.022 (4)	0.022 (4)	-0.003 (4)	0.013 (4)	-0.004 (4)
C20A	0.037 (6)	0.027 (4)	0.015 (4)	-0.013 (4)	0.002 (4)	-0.004 (3)
C21A	0.023 (5)	0.024 (4)	0.019 (4)	-0.009 (4)	0.003 (4)	0.005 (3)
C22A	0.026 (5)	0.023 (4)	0.012 (4)	-0.002 (4)	0.005 (3)	0.003 (3)
C23A	0.015 (5)	0.019 (4)	0.013 (4)	-0.001 (3)	0.001 (3)	-0.002 (3)
C24A	0.019 (5)	0.016 (4)	0.010 (3)	-0.001 (3)	0.002 (3)	-0.001 (3)
C25A	0.011 (4)	0.023 (4)	0.009 (3)	-0.002 (3)	-0.005 (3)	0.002 (3)
O25A	0.022 (3)	0.029 (3)	0.019 (3)	-0.001 (2)	0.007 (2)	0.000 (2)
C26A	0.014 (4)	0.023 (4)	0.011 (4)	-0.001 (3)	0.002 (3)	-0.004 (3)
N27A	0.025 (4)	0.030 (4)	0.016 (3)	0.000 (3)	0.006 (3)	0.004 (3)
N28A	0.013 (4)	0.022 (3)	0.015 (3)	-0.003 (3)	0.002 (3)	-0.002 (3)
C29A	0.018 (5)	0.019 (4)	0.015 (4)	0.001 (3)	0.006 (3)	-0.006 (3)
C30A	0.025 (5)	0.032 (5)	0.017 (4)	-0.001 (4)	0.002 (4)	0.009 (4)
C31A	0.027 (5)	0.029 (4)	0.027 (4)	-0.013 (4)	0.008 (4)	-0.006 (4)

C32A	0.039 (6)	0.021 (4)	0.033 (5)	0.002 (4)	0.014 (4)	0.006 (4)
C33A	0.030 (6)	0.034 (5)	0.024 (4)	0.003 (4)	0.002 (4)	0.009 (4)
C34A	0.019 (5)	0.029 (4)	0.022 (4)	-0.001 (4)	-0.002 (4)	0.002 (4)
C35A	0.017 (5)	0.025 (4)	0.013 (4)	-0.003 (3)	0.006 (3)	0.001 (3)
C36A	0.022 (5)	0.027 (4)	0.022 (4)	-0.001 (4)	0.001 (4)	0.003 (4)
C37A	0.043 (7)	0.025 (4)	0.029 (5)	-0.007 (4)	0.010 (5)	-0.008 (4)
C38A	0.038 (7)	0.027 (5)	0.029 (5)	0.007 (4)	0.011 (4)	-0.001 (4)
C39A	0.026 (5)	0.035 (5)	0.030 (5)	0.005 (4)	0.008 (4)	0.009 (4)
C40A	0.029 (6)	0.028 (4)	0.019 (4)	0.000 (4)	0.007 (4)	-0.001 (3)
Cl1	0.0623 (19)	0.0302 (12)	0.0542 (16)	-0.0043 (12)	0.0298 (14)	-0.0095 (11)
Cl2	0.073 (2)	0.0281 (12)	0.0458 (14)	-0.0002 (12)	0.0289 (14)	0.0047 (11)
Cl3	0.0322 (15)	0.0666 (17)	0.0283 (12)	0.0048 (12)	-0.0017 (10)	-0.0023 (11)
C41	0.041 (6)	0.021 (4)	0.018 (4)	-0.001 (4)	0.007 (4)	0.001 (3)
Cl4	0.0442 (17)	0.0703 (19)	0.0493 (16)	0.0101 (14)	0.0060 (13)	0.0219 (14)
Cl5	0.051 (2)	0.122 (3)	0.0334 (14)	-0.0071 (18)	-0.0032 (13)	0.0108 (16)
Cl6	0.149 (4)	0.0358 (15)	0.083 (2)	0.0037 (18)	0.070 (2)	-0.0027 (15)
C42	0.058 (7)	0.037 (5)	0.021 (4)	0.009 (5)	0.004 (5)	0.005 (4)
Cl7	0.0282 (14)	0.0589 (15)	0.0342 (12)	-0.0018 (11)	0.0012 (10)	0.0165 (11)
Cl8	0.081 (2)	0.0321 (13)	0.0601 (17)	-0.0065 (13)	0.0427 (16)	-0.0128 (12)
Cl9	0.0434 (15)	0.0290 (11)	0.0415 (13)	-0.0023 (10)	0.0140 (11)	0.0053 (10)
C43	0.053 (6)	0.020 (4)	0.015 (4)	-0.001 (4)	0.009 (4)	0.002 (3)
Cl10	0.079 (2)	0.0630 (17)	0.0545 (18)	-0.0035 (14)	0.0136 (15)	0.0044 (13)
Cl11	0.079 (2)	0.0630 (17)	0.0545 (18)	-0.0035 (14)	0.0136 (15)	0.0044 (13)
Cl12	0.079 (2)	0.0630 (17)	0.0545 (18)	-0.0035 (14)	0.0136 (15)	0.0044 (13)
Cl1A	0.093 (6)	0.075 (4)	0.019 (3)	-0.022 (4)	-0.017 (3)	-0.015 (3)
Cl1B	0.093 (6)	0.075 (4)	0.019 (3)	-0.022 (4)	-0.017 (3)	-0.015 (3)
Cl1C	0.093 (6)	0.075 (4)	0.019 (3)	-0.022 (4)	-0.017 (3)	-0.015 (3)
C44	0.082 (9)	0.065 (7)	0.022 (5)	0.005 (7)	0.015 (5)	-0.009 (5)

Geometric parameters (Å, º) for (2a)

C1—N28	1.452 (10)	C5A—C6A	1.366 (12)
C1—C2	1.512 (10)	C5A—H5A	0.9500
C1—C26	1.561 (10)	C6A—C7A	1.386 (11)
C1—H1	1.0000	C6A—H6A	0.9500
C2—C7	1.381 (12)	C7A—O8A	1.370 (9)
C2—C3	1.394 (12)	O8A—C9A	1.398 (10)
C3—C4	1.385 (11)	C9A—C10A	1.500 (12)
C3—H3	0.9500	C9A—H9C	0.9900
C4—C5	1.360 (13)	C9A—H9D	0.9900
C4—H4	0.9500	C10A—N27A	1.319 (10)
C5—C6	1.367 (13)	C10A—C11A	1.396 (12)

C5—H5	0.9500	C11A—C12A	1.369 (13)
C6—C7	1.390 (11)	C11A—H11A	0.9500
C6—H6	0.9500	C12A—C13A	1.373 (13)
C7—O8	1.350 (10)	C12A—H12A	0.9500
O8—C9	1.418 (10)	C13A—C14A	1.386 (13)
C9—C10	1.492 (12)	C13A—H13A	0.9500
C9—H9A	0.9900	C14A—N27A	1.338 (11)
C9—H9B	0.9900	C14A—C15A	1.478 (12)
C10—N27	1.333 (10)	C15A—O16A	1.419 (10)
C10—C11	1.373 (12)	C15A—H15C	0.9900
C11—C12	1.375 (13)	C15A—H15D	0.9900
C11—H11	0.9500	O16A—C17A	1.363 (10)
C12—C13	1.363 (12)	C17A—C22A	1.373 (12)
C12—H12	0.9500	C17A—C18A	1.394 (11)
C13—C14	1.398 (12)	C18A—C19A	1.358 (12)
C13—H13	0.9500	C18A—H18A	0.9500
C14—N27	1.333 (11)	C19A—C20A	1.363 (13)
C14—C15	1.481 (12)	C19A—H19A	0.9500
C15—O16	1.421 (10)	C20A—C21A	1.383 (11)
C15—H15A	0.9900	C20A—H20A	0.9500
C15—H15B	0.9900	C21A—C22A	1.385 (11)
O16—C17	1.360 (10)	C21A—H21A	0.9500
C17—C22	1.373 (12)	C22A—C23A	1.509 (10)
C17—C18	1.406 (11)	C23A—N28A	1.459 (10)
C18—C19	1.370 (12)	C23A—C24A	1.565 (9)
C18—H18	0.9500	C23A—H23A	1.0000
C19—C20	1.366 (13)	C24A—C29A	1.502 (10)
C19—H19	0.9500	C24A—C25A	1.519 (10)
C20—C21	1.389 (12)	C24A—H24A	1.0000
C20—H20	0.9500	C25A—O25A	1.218 (9)
C21—C22	1.387 (11)	C25A—C26A	1.509 (10)
C21—H21	0.9500	C26A—C35A	1.503 (11)
C22—C23	1.508 (10)	C26A—H26A	1.0000
C23—N28	1.455 (9)	N28A—H28A	0.78 (9)
C23—C24	1.558 (10)	C29A—C30A	1.369 (12)
C23—H23	1.0000	C29A—C34A	1.384 (11)
C24—C29	1.500 (10)	C30A—C31A	1.384 (11)
C24—C25	1.511 (11)	C30A—H30A	0.9500
C24—H24	1.0000	C31A—C32A	1.382 (12)
C25—O25	1.213 (9)	C31A—H31A	0.9500
C25—C26	1.508 (10)	C32A—C33A	1.366 (13)

C26—C35	1.506 (11)	C32A—H32A	0.9500
C26—H26	1.0000	C33A—C34A	1.379 (12)
N28—H28	0.81 (9)	C33A—H33A	0.9500
C29—C30	1.369 (12)	C34A—H34A	0.9500
C29—C34	1.392 (11)	C35A—C40A	1.364 (12)
C30—C31	1.386 (12)	C35A—C36A	1.381 (11)
C30—H30	0.9500	C36A—C37A	1.385 (12)
C31—C32	1.380 (13)	C36A—H36A	0.9500
C31—H31	0.9500	C37A—C38A	1.383 (13)
C32—C33	1.361 (13)	C37A—H37A	0.9500
C32—H32	0.9500	C38A—C39A	1.372 (13)
C33—C34	1.376 (12)	C38A—H38A	0.9500
C33—H33	0.9500	C39A—C40A	1.387 (12)
C34—H34	0.9500	C39A—H39A	0.9500
C35—C40	1.373 (12)	C40A—H40A	0.9500
C35—C36	1.373 (11)	Cl1—C41	1.752 (8)
C36—C37	1.383 (12)	Cl2—C41	1.752 (8)
C36—H36	0.9500	Cl3—C41	1.733 (9)
C37—C38	1.363 (14)	C41—H41	1.0000
C37—H37	0.9500	Cl4—C42	1.746 (9)
C38—C39	1.368 (13)	Cl5—C42	1.720 (11)
C38—H38	0.9500	Cl6—C42	1.736 (10)
C39—C40	1.381 (12)	C42—H42	1.0000
C39—H39	0.9500	Cl7—C43	1.738 (10)
C40—H40	0.9500	Cl8—C43	1.749 (8)
C1A—N28A	1.447 (10)	Cl9—C43	1.752 (8)
C1A—C2A	1.525 (10)	C43—H43	1.0000
C1A—C26A	1.565 (9)	Cl10—C44	1.756 (3)
C1A—H1A	1.0000	Cl11—C44	1.771 (3)
C2A—C3A	1.377 (11)	Cl12—C44	1.750 (3)
C2A—C7A	1.379 (11)	Cl1A—C44	1.760 (3)
C3A—C4A	1.398 (12)	Cl1B—C44	1.749 (3)
C3A—H3A	0.9500	Cl1C—C44	1.770 (3)
C4A—C5A	1.362 (13)	C44—H44A	1.0000
C4A—H4A	0.9500	C44—H44B	1.0000
N28—C1—C2	113.0 (6)	C4A—C5A—C6A	121.4 (8)
N28—C1—C26	111.3 (6)	C4A—C5A—H5A	119.3
C2—C1—C26	114.3 (6)	C6A—C5A—H5A	119.3
N28—C1—H1	105.8	C5A—C6A—C7A	119.8 (8)
C2—C1—H1	105.8	C5A—C6A—H6A	120.1

C26—C1—H1	105.8	C7A—C6A—H6A	120.1
C7—C2—C3	118.3 (7)	O8A—C7A—C2A	116.6 (7)
C7—C2—C1	122.7 (7)	O8A—C7A—C6A	123.3 (7)
C3—C2—C1	119.0 (7)	C2A—C7A—C6A	120.2 (8)
C4—C3—C2	120.6 (8)	C7A—O8A—C9A	118.7 (6)
C4—C3—H3	119.7	O8A—C9A—C10A	108.4 (6)
C2—C3—H3	119.7	O8A—C9A—H9C	110.0
C5—C4—C3	119.7 (8)	C10A—C9A—H9C	110.0
C5—C4—H4	120.2	O8A—C9A—H9D	110.0
C3—C4—H4	120.2	C10A—C9A—H9D	110.0
C4—C5—C6	121.2 (8)	H9C—C9A—H9D	108.4
C4—C5—H5	119.4	N27A—C10A—C11A	122.9 (8)
C6—C5—H5	119.4	N27A—C10A—C9A	114.9 (7)
C5—C6—C7	119.4 (8)	C11A—C10A—C9A	122.1 (8)
C5—C6—H6	120.3	C12A—C11A—C10A	118.6 (8)
C7—C6—H6	120.3	C12A—C11A—H11A	120.7
O8—C7—C2	116.0 (7)	C10A—C11A—H11A	120.7
O8—C7—C6	123.3 (8)	C11A—C12A—C13A	119.0 (8)
C2—C7—C6	120.7 (8)	C11A—C12A—H12A	120.5
C7—O8—C9	117.3 (6)	C13A—C12A—H12A	120.5
O8—C9—C10	107.4 (7)	C12A—C13A—C14A	118.7 (8)
O8—C9—H9A	110.2	C12A—C13A—H13A	120.6
C10—C9—H9A	110.2	C14A—C13A—H13A	120.6
O8—C9—H9B	110.2	N27A—C14A—C13A	122.8 (8)
C10—C9—H9B	110.2	N27A—C14A—C15A	115.7 (8)
H9A—C9—H9B	108.5	C13A—C14A—C15A	121.5 (8)
N27—C10—C11	122.9 (8)	O16A—C15A—C14A	106.6 (7)
N27—C10—C9	115.0 (7)	O16A—C15A—H15C	110.4
C11—C10—C9	122.1 (8)	C14A—C15A—H15C	110.4
C10—C11—C12	119.4 (9)	O16A—C15A—H15D	110.4
C10—C11—H11	120.3	C14A—C15A—H15D	110.4
C12—C11—H11	120.3	H15C—C15A—H15D	108.6
C13—C12—C11	118.6 (8)	C17A—O16A—C15A	118.0 (6)
C13—C12—H12	120.7	O16A—C17A—C22A	116.3 (7)
C11—C12—H12	120.7	O16A—C17A—C18A	122.5 (8)
C12—C13—C14	119.0 (8)	C22A—C17A—C18A	121.3 (8)
C12—C13—H13	120.5	C19A—C18A—C17A	119.5 (8)
C14—C13—H13	120.5	C19A—C18A—H18A	120.3
N27—C14—C13	122.4 (8)	C17A—C18A—H18A	120.3
N27—C14—C15	115.5 (7)	C18A—C19A—C20A	121.1 (8)
C13—C14—C15	122.0 (8)	C18A—C19A—H19A	119.4

O16—C15—C14	108.3 (7)	C20A—C19A—H19A	119.4
O16—C15—H15A	110.0	C19A—C20A—C21A	118.8 (8)
C14—C15—H15A	110.0	C19A—C20A—H20A	120.6
O16—C15—H15B	110.0	C21A—C20A—H20A	120.6
C14—C15—H15B	110.0	C20A—C21A—C22A	122.0 (8)
H15A—C15—H15B	108.4	C20A—C21A—H21A	119.0
C17—O16—C15	119.3 (6)	C22A—C21A—H21A	119.0
O16—C17—C22	116.9 (7)	C17A—C22A—C21A	117.3 (7)
O16—C17—C18	122.5 (8)	C17A—C22A—C23A	122.8 (7)
C22—C17—C18	120.6 (8)	C21A—C22A—C23A	119.8 (7)
C19—C18—C17	119.6 (8)	N28A—C23A—C22A	112.8 (6)
C19—C18—H18	120.2	N28A—C23A—C24A	110.7 (6)
C17—C18—H18	120.2	C22A—C23A—C24A	115.0 (6)
C20—C19—C18	120.5 (8)	N28A—C23A—H23A	105.9
C20—C19—H19	119.8	C22A—C23A—H23A	105.9
C18—C19—H19	119.8	C24A—C23A—H23A	105.9
C19—C20—C21	119.8 (8)	C29A—C24A—C25A	111.7 (6)
C19—C20—H20	120.1	C29A—C24A—C23A	115.0 (6)
C21—C20—H20	120.1	C25A—C24A—C23A	106.1 (6)
C22—C21—C20	120.9 (8)	C29A—C24A—H24A	107.9
C22—C21—H21	119.5	C25A—C24A—H24A	107.9
C20—C21—H21	119.5	C23A—C24A—H24A	107.9
C17—C22—C21	118.6 (7)	O25A—C25A—C26A	122.2 (7)
C17—C22—C23	122.3 (7)	O25A—C25A—C24A	120.8 (7)
C21—C22—C23	119.1 (7)	C26A—C25A—C24A	117.0 (6)
N28—C23—C22	112.8 (6)	C35A—C26A—C25A	113.6 (6)
N28—C23—C24	112.6 (6)	C35A—C26A—C1A	111.3 (6)
C22—C23—C24	113.9 (6)	C25A—C26A—C1A	109.4 (6)
N28—C23—H23	105.6	C35A—C26A—H26A	107.4
C22—C23—H23	105.6	C25A—C26A—H26A	107.4
C24—C23—H23	105.6	C1A—C26A—H26A	107.4
C29—C24—C25	113.5 (6)	C10A—N27A—C14A	117.9 (7)
C29—C24—C23	112.5 (6)	C1A—N28A—C23A	109.9 (6)
C25—C24—C23	108.6 (6)	C1A—N28A—H28A	112 (6)
C29—C24—H24	107.3	C23A—N28A—H28A	111 (6)
C25—C24—H24	107.3	C30A—C29A—C34A	117.8 (7)
C23—C24—H24	107.3	C30A—C29A—C24A	122.4 (7)
O25—C25—C26	122.1 (7)	C34A—C29A—C24A	119.7 (7)
O25—C25—C24	122.3 (7)	C29A—C30A—C31A	121.4 (8)
C26—C25—C24	115.6 (6)	C29A—C30A—H30A	119.3
C35—C26—C25	113.6 (6)	C31A—C30A—H30A	119.3

C35—C26—C1	113.6 (6)	C32A—C31A—C30A	119.7 (8)
C25—C26—C1	107.1 (6)	C32A—C31A—H31A	120.1
C35—C26—H26	107.4	C30A—C31A—H31A	120.1
C25—C26—H26	107.4	C33A—C32A—C31A	119.6 (8)
C1—C26—H26	107.4	C33A—C32A—H32A	120.2
C14—N27—C10	117.7 (7)	C31A—C32A—H32A	120.2
C1—N28—C23	110.4 (6)	C32A—C33A—C34A	119.9 (8)
C1—N28—H28	115 (6)	C32A—C33A—H33A	120.1
C23—N28—H28	105 (6)	C34A—C33A—H33A	120.1
C30—C29—C34	118.0 (7)	C33A—C34A—C29A	121.5 (8)
C30—C29—C24	121.4 (7)	C33A—C34A—H34A	119.3
C34—C29—C24	120.6 (7)	C29A—C34A—H34A	119.3
C29—C30—C31	121.2 (8)	C40A—C35A—C36A	119.1 (8)
C29—C30—H30	119.4	C40A—C35A—C26A	120.3 (7)
C31—C30—H30	119.4	C36A—C35A—C26A	120.5 (7)
C32—C31—C30	119.5 (9)	C35A—C36A—C37A	120.2 (8)
C32—C31—H31	120.2	C35A—C36A—H36A	119.9
C30—C31—H31	120.2	C37A—C36A—H36A	119.9
C33—C32—C31	120.1 (8)	C38A—C37A—C36A	120.4 (8)
C33—C32—H32	120.0	C38A—C37A—H37A	119.8
C31—C32—H32	120.0	C36A—C37A—H37A	119.8
C32—C33—C34	120.1 (9)	C39A—C38A—C37A	119.0 (8)
C32—C33—H33	120.0	C39A—C38A—H38A	120.5
C34—C33—H33	120.0	C37A—C38A—H38A	120.5
C33—C34—C29	121.1 (8)	C38A—C39A—C40A	120.2 (9)
C33—C34—H34	119.5	C38A—C39A—H39A	119.9
C29—C34—H34	119.5	C40A—C39A—H39A	119.9
C40—C35—C36	117.6 (8)	C35A—C40A—C39A	121.1 (8)
C40—C35—C26	122.3 (7)	C35A—C40A—H40A	119.5
C36—C35—C26	120.1 (7)	C39A—C40A—H40A	119.5
C35—C36—C37	121.7 (9)	Cl3—C41—Cl1	110.5 (5)
C35—C36—H36	119.1	Cl3—C41—Cl2	111.3 (5)
C37—C36—H36	119.1	Cl1—C41—Cl2	109.5 (4)
C38—C37—C36	120.1 (9)	Cl3—C41—H41	108.5
C38—C37—H37	120.0	Cl1—C41—H41	108.5
C36—C37—H37	120.0	Cl2—C41—H41	108.5
C37—C38—C39	118.9 (8)	Cl5—C42—Cl6	111.2 (6)
C37—C38—H38	120.6	Cl5—C42—Cl4	109.7 (5)
C39—C38—H38	120.6	Cl6—C42—Cl4	110.0 (5)
C38—C39—C40	120.9 (9)	Cl5—C42—H42	108.6
C38—C39—H39	119.6	Cl6—C42—H42	108.6

C40—C39—H39	119.6	Cl4—C42—H42	108.6
C35—C40—C39	120.9 (8)	Cl7—C43—Cl8	110.6 (5)
C35—C40—H40	119.6	Cl7—C43—Cl9	110.6 (5)
C39—C40—H40	119.6	Cl8—C43—Cl9	109.4 (4)
N28A—C1A—C2A	113.6 (6)	Cl7—C43—H43	108.8
N28A—C1A—C26A	113.7 (6)	Cl8—C43—H43	108.8
C2A—C1A—C26A	110.9 (6)	Cl9—C43—H43	108.8
N28A—C1A—H1A	106.0	Cl12—C44—Cl10	110.7 (2)
C2A—C1A—H1A	106.0	Cl1B—C44—Cl1A	110.5 (2)
C26A—C1A—H1A	106.0	Cl1B—C44—Cl1C	110.1 (2)
C3A—C2A—C7A	119.0 (7)	Cl1A—C44—Cl1C	108.8 (2)
C3A—C2A—C1A	118.9 (7)	Cl12—C44—Cl11	109.6 (2)
C7A—C2A—C1A	122.0 (7)	Cl10—C44—Cl11	109.0 (2)
C2A—C3A—C4A	121.0 (8)	Cl12—C44—H44A	109.2
C2A—C3A—H3A	119.5	Cl10—C44—H44A	109.2
C4A—C3A—H3A	119.5	Cl11—C44—H44A	109.2
C5A—C4A—C3A	118.5 (8)	Cl1B—C44—H44B	109.1
C5A—C4A—H4A	120.7	Cl1A—C44—H44B	109.1
C3A—C4A—H4A	120.7	Cl1C—C44—H44B	109.1
N28—C1—C2—C7	64.8 (10)	N28A—C1A—C2A—C3A	-117.8 (8)
C26—C1—C2—C7	-63.8 (10)	C26A—C1A—C2A—C3A	112.7 (8)
N28—C1—C2—C3	-114.3 (8)	N28A—C1A—C2A—C7A	63.8 (9)
C26—C1—C2—C3	117.0 (8)	C26A—C1A—C2A—C7A	-65.7 (9)
C7—C2—C3—C4	-1.0 (11)	C7A—C2A—C3A—C4A	0.7 (11)
C1—C2—C3—C4	178.2 (7)	C1A—C2A—C3A—C4A	-177.7 (7)
C2—C3—C4—C5	-0.1 (12)	C2A—C3A—C4A—C5A	-1.1 (12)
C3—C4—C5—C6	1.0 (12)	C3A—C4A—C5A—C6A	0.5 (12)
C4—C5—C6—C7	-0.6 (12)	C4A—C5A—C6A—C7A	0.4 (12)
C3—C2—C7—O8	-179.4 (7)	C3A—C2A—C7A—O8A	-179.0 (6)
C1—C2—C7—O8	1.4 (11)	C1A—C2A—C7A—O8A	-0.6 (10)
C3—C2—C7—C6	1.4 (11)	C3A—C2A—C7A—C6A	0.2 (11)
C1—C2—C7—C6	-177.8 (7)	C1A—C2A—C7A—C6A	178.6 (7)
C5—C6—C7—O8	-179.7 (7)	C5A—C6A—C7A—O8A	178.4 (7)
C5—C6—C7—C2	-0.6 (12)	C5A—C6A—C7A—C2A	-0.7 (11)
C2—C7—O8—C9	-154.6 (7)	C2A—C7A—O8A—C9A	-162.3 (6)
C6—C7—O8—C9	24.6 (10)	C6A—C7A—O8A—C9A	18.6 (10)
C7—O8—C9—C10	158.5 (6)	C7A—O8A—C9A—C10A	152.2 (6)
O8—C9—C10—N27	-49.6 (9)	O8A—C9A—C10A—N27A	-40.3 (9)
O8—C9—C10—C11	131.8 (8)	O8A—C9A—C10A—C11A	143.1 (7)
N27—C10—C11—C12	0.6 (13)	N27A—C10A—C11A—C12A	-1.2 (12)

C9—C10—C11—C12	179.1 (8)	C9A—C10A—C11A—C12A	175.1 (7)
C10—C11—C12—C13	-0.2 (12)	C10A—C11A—C12A—C13A	2.2 (12)
C11—C12—C13—C14	0.3 (12)	C11A—C12A—C13A—C14A	-1.1 (12)
C12—C13—C14—N27	-0.9 (12)	C12A—C13A—C14A—N27A	-1.1 (12)
C12—C13—C14—C15	-177.0 (8)	C12A—C13A—C14A—C15A	-178.8 (8)
N27—C14—C15—O16	43.8 (9)	N27A—C14A—C15A—O16A	47.2 (9)
C13—C14—C15—O16	-139.8 (7)	C13A—C14A—C15A—O16A	-134.9 (8)
C14—C15—O16—C17	-149.3 (7)	C14A—C15A—O16A—C17A	-158.6 (6)
C15—O16—C17—C22	159.6 (7)	C15A—O16A—C17A—C22A	157.3 (7)
C15—O16—C17—C18	-21.1 (10)	C15A—O16A—C17A—C18A	-22.7 (10)
O16—C17—C18—C19	179.5 (7)	O16A—C17A—C18A—C19A	-179.7 (7)
C22—C17—C18—C19	-1.2 (12)	C22A—C17A—C18A—C19A	0.2 (12)
C17—C18—C19—C20	0.5 (12)	C17A—C18A—C19A—C20A	0.5 (12)
C18—C19—C20—C21	0.3 (13)	C18A—C19A—C20A—C21A	-0.8 (12)
C19—C20—C21—C22	-0.2 (12)	C19A—C20A—C21A—C22A	0.5 (12)
O16—C17—C22—C21	-179.4 (6)	O16A—C17A—C22A—C21A	179.4 (6)
C18—C17—C22—C21	1.3 (11)	C18A—C17A—C22A—C21A	-0.5 (11)
O16—C17—C22—C23	-1.5 (10)	O16A—C17A—C22A—C23A	-3.3 (10)
C18—C17—C22—C23	179.2 (7)	C18A—C17A—C22A—C23A	176.7 (7)
C20—C21—C22—C17	-0.6 (11)	C20A—C21A—C22A—C17A	0.1 (11)
C20—C21—C22—C23	-178.6 (7)	C20A—C21A—C22A—C23A	-177.2 (7)
C17—C22—C23—N28	-67.2 (9)	C17A—C22A—C23A—N28A	-64.1 (9)
C21—C22—C23—N28	110.7 (8)	C21A—C22A—C23A—N28A	113.1 (8)
C17—C22—C23—C24	62.8 (9)	C17A—C22A—C23A—C24A	64.1 (10)
C21—C22—C23—C24	-119.3 (7)	C21A—C22A—C23A—C24A	-118.7 (8)
N28—C23—C24—C29	-178.6 (6)	N28A—C23A—C24A—C29A	176.0 (6)
C22—C23—C24—C29	51.4 (9)	C22A—C23A—C24A—C29A	46.7 (9)
N28—C23—C24—C25	-52.1 (8)	N28A—C23A—C24A—C25A	-60.0 (8)
C22—C23—C24—C25	177.9 (6)	C22A—C23A—C24A—C25A	170.7 (6)
C29—C24—C25—O25	-3.2 (10)	C29A—C24A—C25A—O25A	-1.1 (10)
C23—C24—C25—O25	-129.2 (8)	C23A—C24A—C25A—O25A	-127.1 (7)
C29—C24—C25—C26	176.4 (7)	C29A—C24A—C25A—C26A	177.8 (6)
C23—C24—C25—C26	50.5 (8)	C23A—C24A—C25A—C26A	51.8 (8)
O25—C25—C26—C35	0.4 (11)	O25A—C25A—C26A—C35A	7.9 (10)
C24—C25—C26—C35	-179.3 (7)	C24A—C25A—C26A—C35A	-170.9 (6)
O25—C25—C26—C1	126.6 (8)	O25A—C25A—C26A—C1A	133.0 (7)
C24—C25—C26—C1	-53.0 (9)	C24A—C25A—C26A—C1A	-45.9 (9)
N28—C1—C26—C35	-176.0 (6)	N28A—C1A—C26A—C35A	173.9 (6)
C2—C1—C26—C35	-46.5 (10)	C2A—C1A—C26A—C35A	-56.6 (8)
N28—C1—C26—C25	57.7 (8)	N28A—C1A—C26A—C25A	47.6 (8)
C2—C1—C26—C25	-172.8 (7)	C2A—C1A—C26A—C25A	177.0 (6)

C13—C14—N27—C10	1.2 (11)	C11A—C10A—N27A—C14A	-0.9 (11)
C15—C14—N27—C10	177.6 (7)	C9A—C10A—N27A—C14A	-177.4 (7)
C11—C10—N27—C14	-1.1 (11)	C13A—C14A—N27A—C10A	2.1 (11)
C9—C10—N27—C14	-179.6 (7)	C15A—C14A—N27A—C10A	179.9 (7)
C2—C1—N28—C23	167.0 (6)	C2A—C1A—N28A—C23A	173.0 (6)
C26—C1—N28—C23	-62.9 (8)	C26A—C1A—N28A—C23A	-58.9 (8)
C22—C23—N28—C1	-169.5 (6)	C22A—C23A—N28A—C1A	-163.8 (6)
C24—C23—N28—C1	59.9 (8)	C24A—C23A—N28A—C1A	65.8 (8)
C25—C24—C29—C30	-62.2 (9)	C25A—C24A—C29A—C30A	-79.5 (9)
C23—C24—C29—C30	61.6 (9)	C23A—C24A—C29A—C30A	41.5 (10)
C25—C24—C29—C34	117.6 (8)	C25A—C24A—C29A—C34A	98.4 (8)
C23—C24—C29—C34	-118.6 (8)	C23A—C24A—C29A—C34A	-140.6 (7)
C34—C29—C30—C31	1.0 (12)	C34A—C29A—C30A—C31A	0.0 (12)
C24—C29—C30—C31	-179.1 (8)	C24A—C29A—C30A—C31A	178.0 (7)
C29—C30—C31—C32	-0.4 (13)	C29A—C30A—C31A—C32A	0.9 (13)
C30—C31—C32—C33	0.2 (14)	C30A—C31A—C32A—C33A	-0.9 (13)
C31—C32—C33—C34	-0.5 (14)	C31A—C32A—C33A—C34A	0.1 (13)
C32—C33—C34—C29	1.1 (13)	C32A—C33A—C34A—C29A	0.8 (13)
C30—C29—C34—C33	-1.4 (12)	C30A—C29A—C34A—C33A	-0.9 (12)
C24—C29—C34—C33	178.8 (7)	C24A—C29A—C34A—C33A	-178.9 (7)
C25—C26—C35—C40	72.9 (9)	C25A—C26A—C35A—C40A	62.4 (9)
C1—C26—C35—C40	-49.9 (10)	C1A—C26A—C35A—C40A	-61.7 (9)
C25—C26—C35—C36	-109.5 (8)	C25A—C26A—C35A—C36A	-119.3 (8)
C1—C26—C35—C36	127.7 (8)	C1A—C26A—C35A—C36A	116.7 (8)
C40—C35—C36—C37	1.6 (12)	C40A—C35A—C36A—C37A	2.4 (11)
C26—C35—C36—C37	-176.1 (7)	C26A—C35A—C36A—C37A	-176.0 (7)
C35—C36—C37—C38	-0.9 (13)	C35A—C36A—C37A—C38A	-1.7 (12)
C36—C37—C38—C39	-0.4 (14)	C36A—C37A—C38A—C39A	0.2 (13)
C37—C38—C39—C40	0.8 (13)	C37A—C38A—C39A—C40A	0.6 (13)
C36—C35—C40—C39	-1.2 (12)	C36A—C35A—C40A—C39A	-1.6 (11)
C26—C35—C40—C39	176.5 (7)	C26A—C35A—C40A—C39A	176.8 (7)
C38—C39—C40—C35	0.0 (13)	C38A—C39A—C40A—C35A	0.1 (12)

References

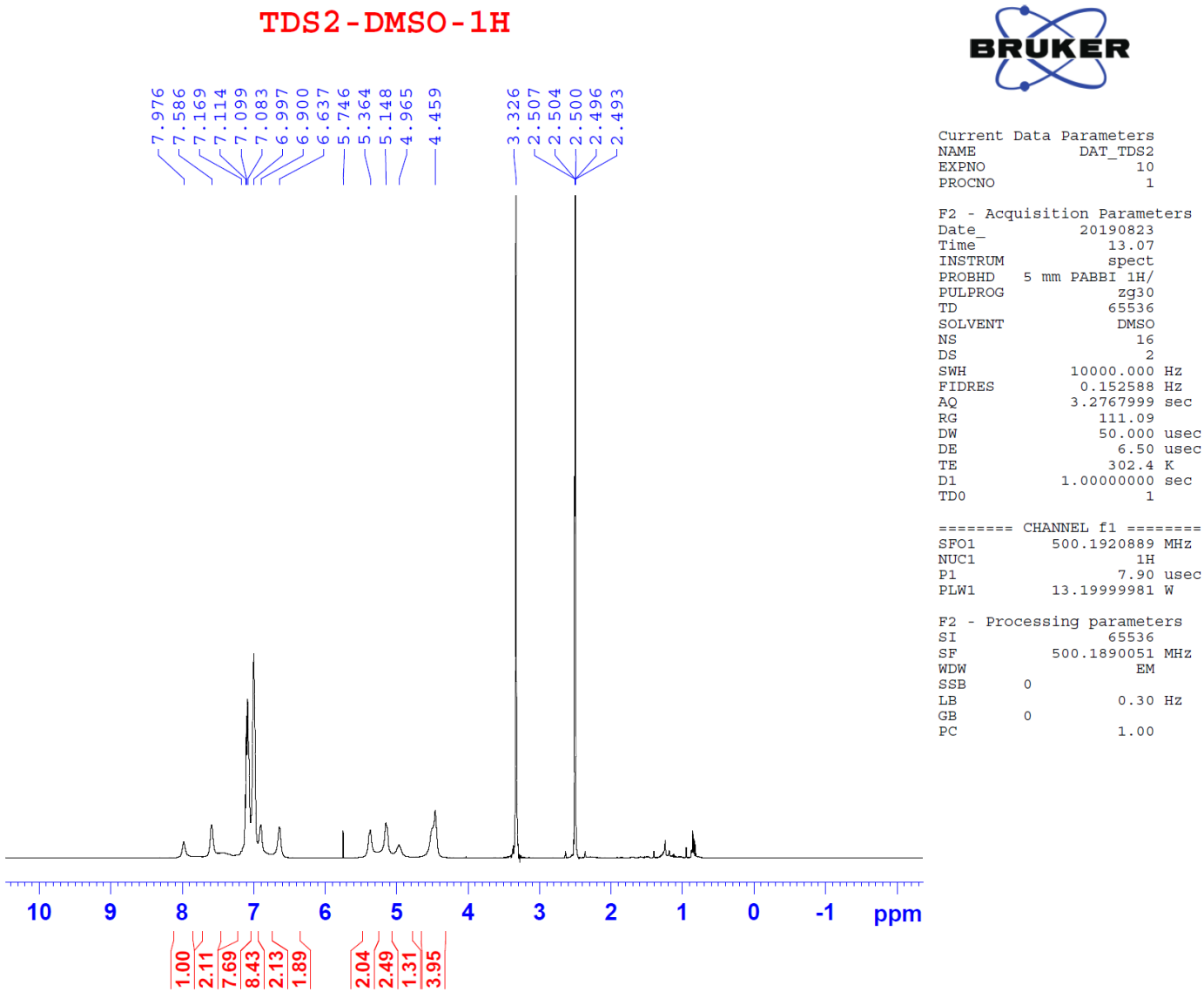
[S1] N. A. Bailey, D. E. Fenton, S. J. Kitchen, T. H. Lilley. M. G. Williams, P. A. Tasker, A. J. Leong and L. F. Lindoy, *J. Chem. Soc., Dalton Trans.*, 1991, 627.

[S2] P. Skehan, R. Storeng, D. Scudiero, A. Monks, J. McMahon, D. Vistica, J. T. Warren, H. Bokesch, S. Kenney and M. R. Boyd, *J. Natl. Cancer Inst.* 1990, **82**, 1107.

[S3] K. Likhitwitayawuid, C. K. Angerhofer, G. A. Cordell, J. M. Pezzuto and N. Ruangrungsi, *J. Nat. Prod.*, 1993, **56**, 30.

[S4] D. A. Vanden Berghe and A. J. Vlietlinck, in *Methods in Plant Biochemistry*, ed. K. Hostettmann, Academic Press, London, 1991, vol. 6, pp. 47-59.

[S5] L. McKane and J. Kandel, *Microbiology: Essentials and Applications*, McGraw-Hill, New York, 1996.

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TD                65536
SOLVENT            DMSO
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG               111.09
DW                50.000 usec
DE                 6.50 usec
TE                302.4 K
D1            1.00000000 sec
TD0                   1

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NUC1                1H
P1                 7.90 usec
PLW1        13.19999981 W

F2 - Processing parameters
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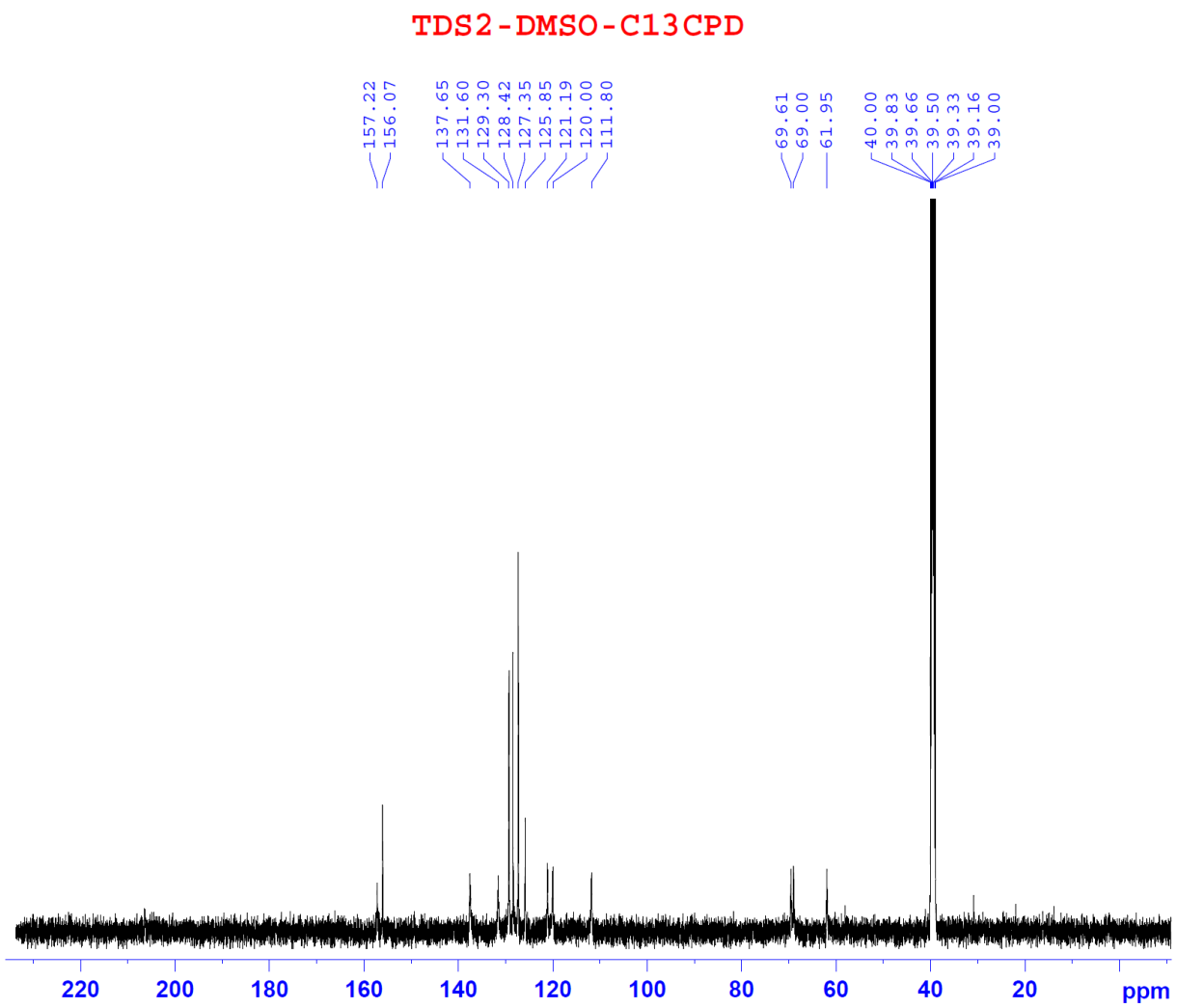
Picture S1. *^{1}H-NMR spectrum of compound **2a***

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PROCNO                       1

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TD                       65536
SOLVENT                  DMSO
NS                       3072
DS                          4
SWH                 31250.000 Hz
FIDRES               0.476837 Hz
AQ                   1.0485760 sec
RG                      198.57
DW                      16.000 usec
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TE                      304.8 K
D1                   2.00000000 sec
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TD0                          1

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NUC1                       13C
P1                       10.00 usec
PLW1                88.00000000 W

======== CHANNEL f2 ========
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NUC2                       1H
CPDPRG[2                waltz16
PCPD2                    80.00 usec
PLW2                22.00000000 W
PLW12                0.35764000 W
PLW13                0.17989001 W

F2 - Processing parameters
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SF                  125.7726926 MHz
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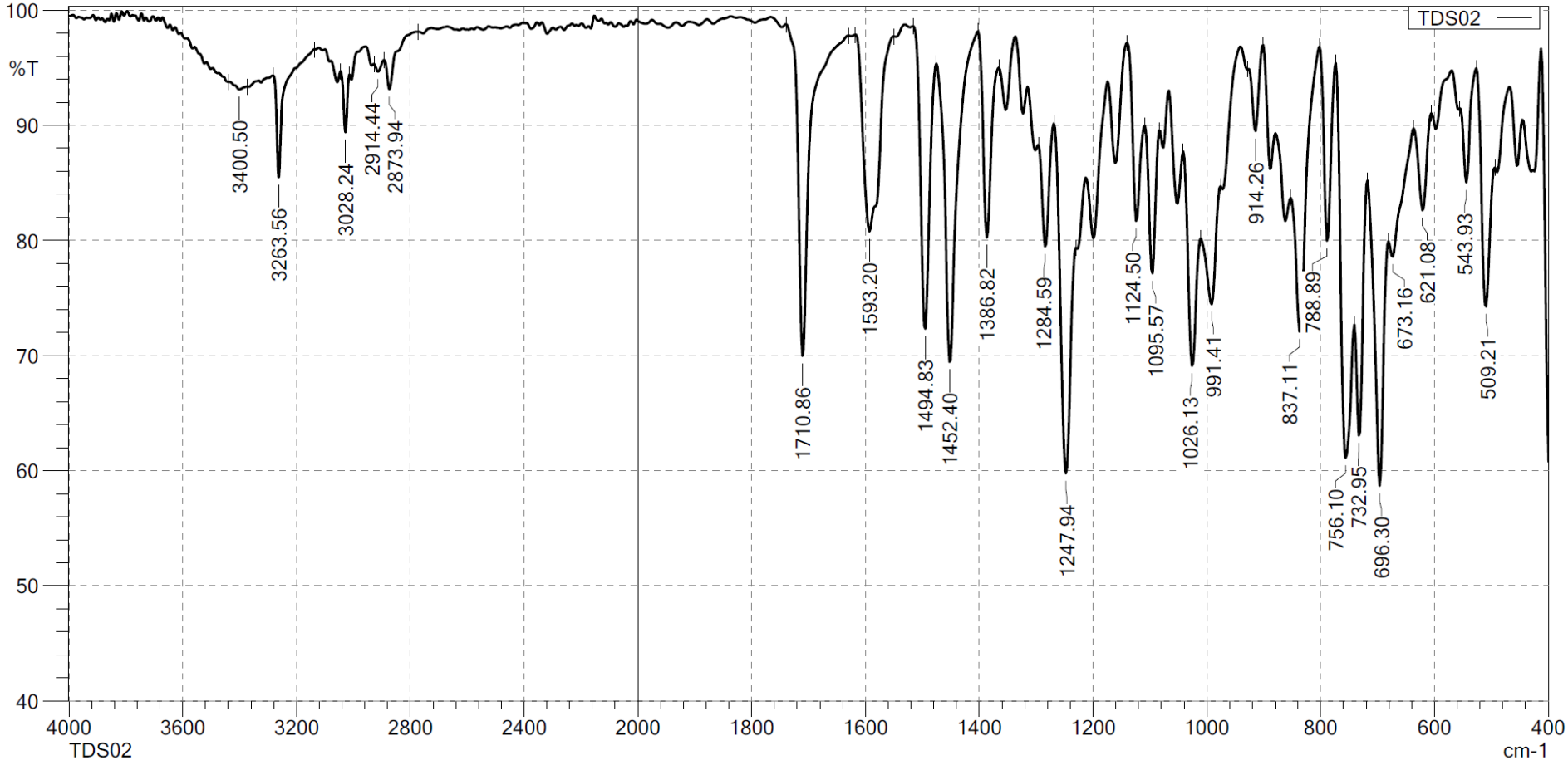
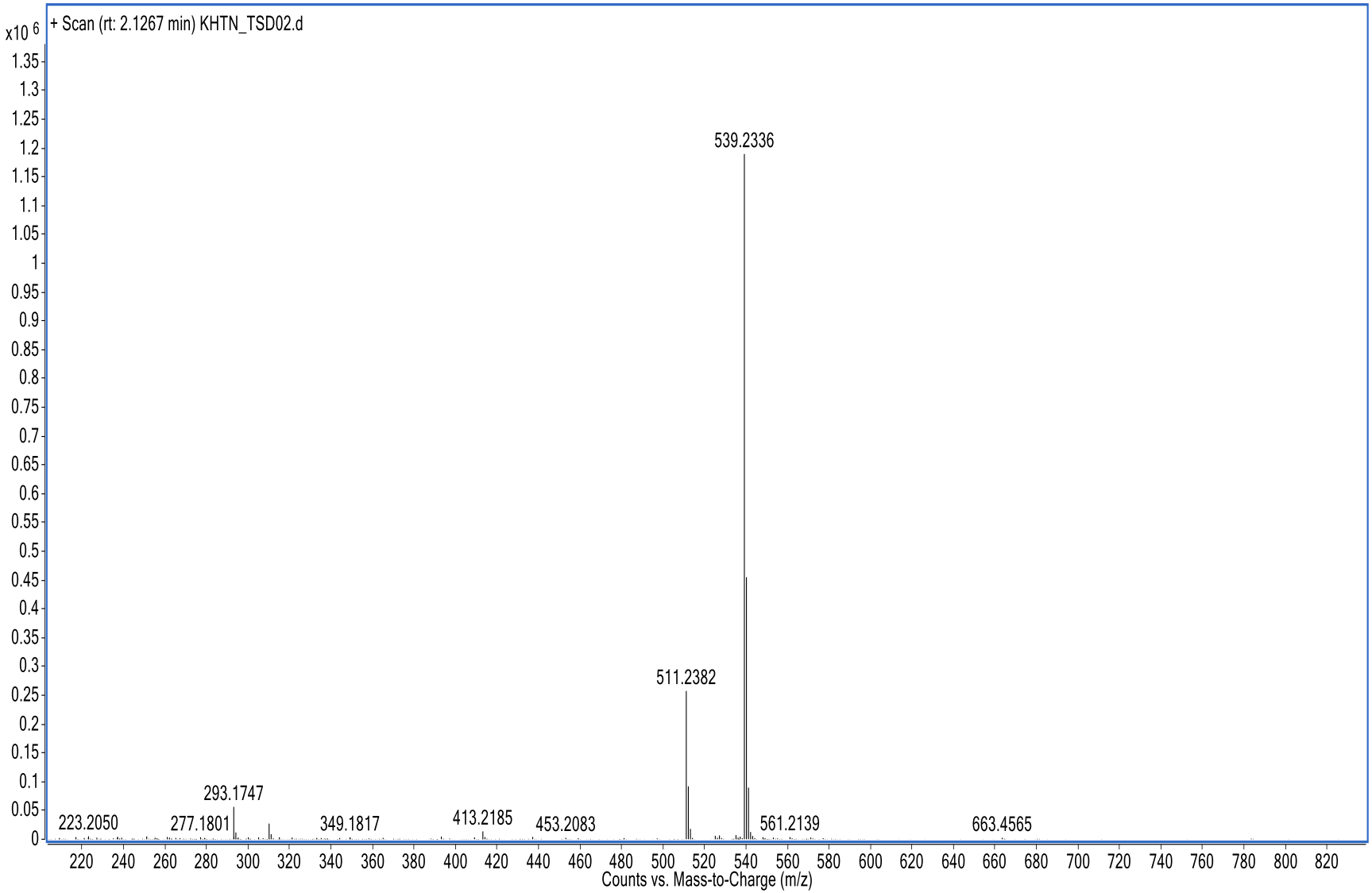
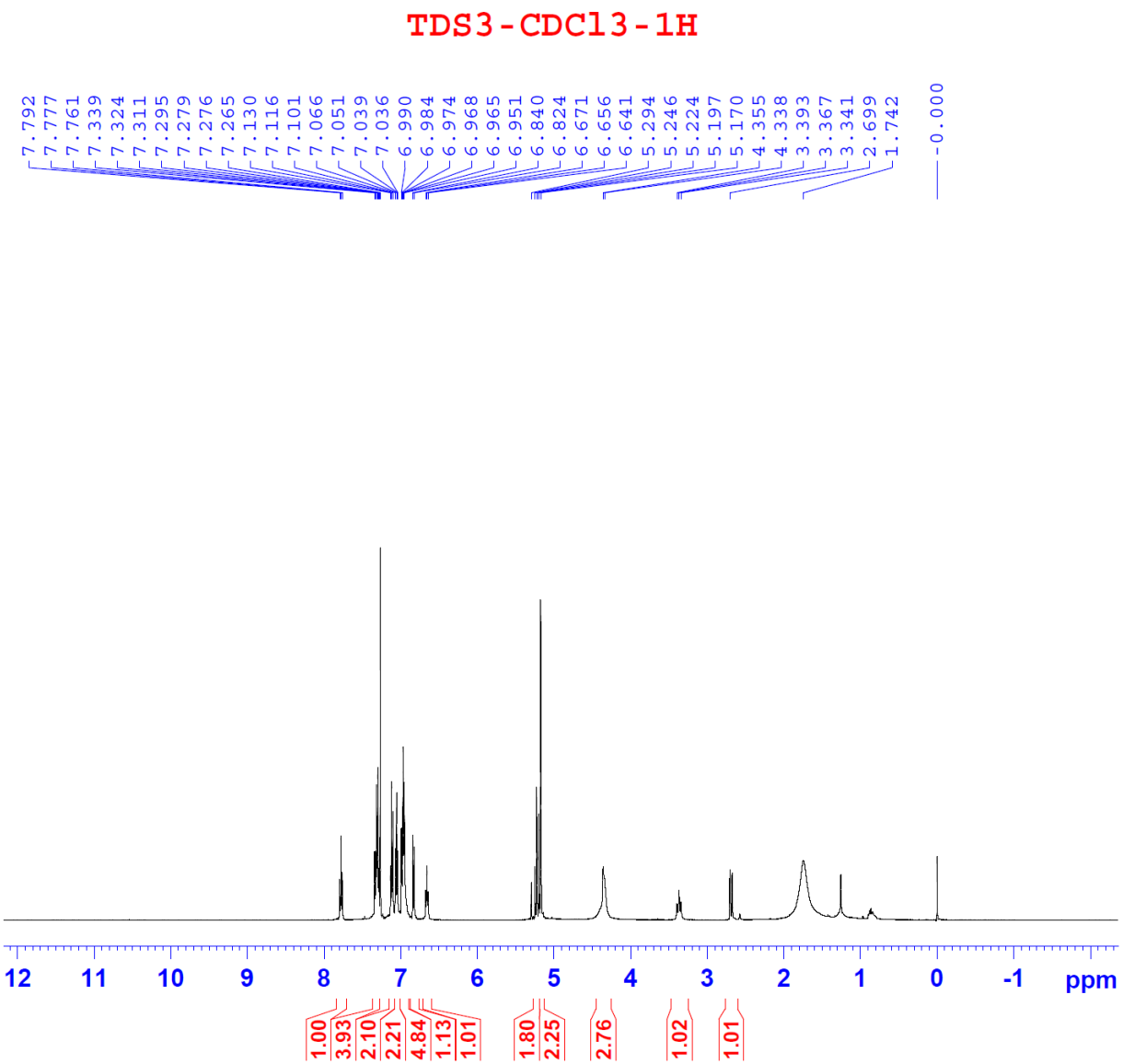
Picture S2. *^{13}C NMR spectrum of compound **2a***



Picture S3. *IR Spectrum of compound* ***2a***



Picture S4. *HRMS spectrum of compound* ***2a***

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PROCNO                 1

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SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG               198.57
DW                50.000 usec
DE                 6.50 usec
TE                302.4 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1920889 MHz
NUC1                1H
P1                 7.90 usec
PLW1        13.19999981 W

F2 - Processing parameters
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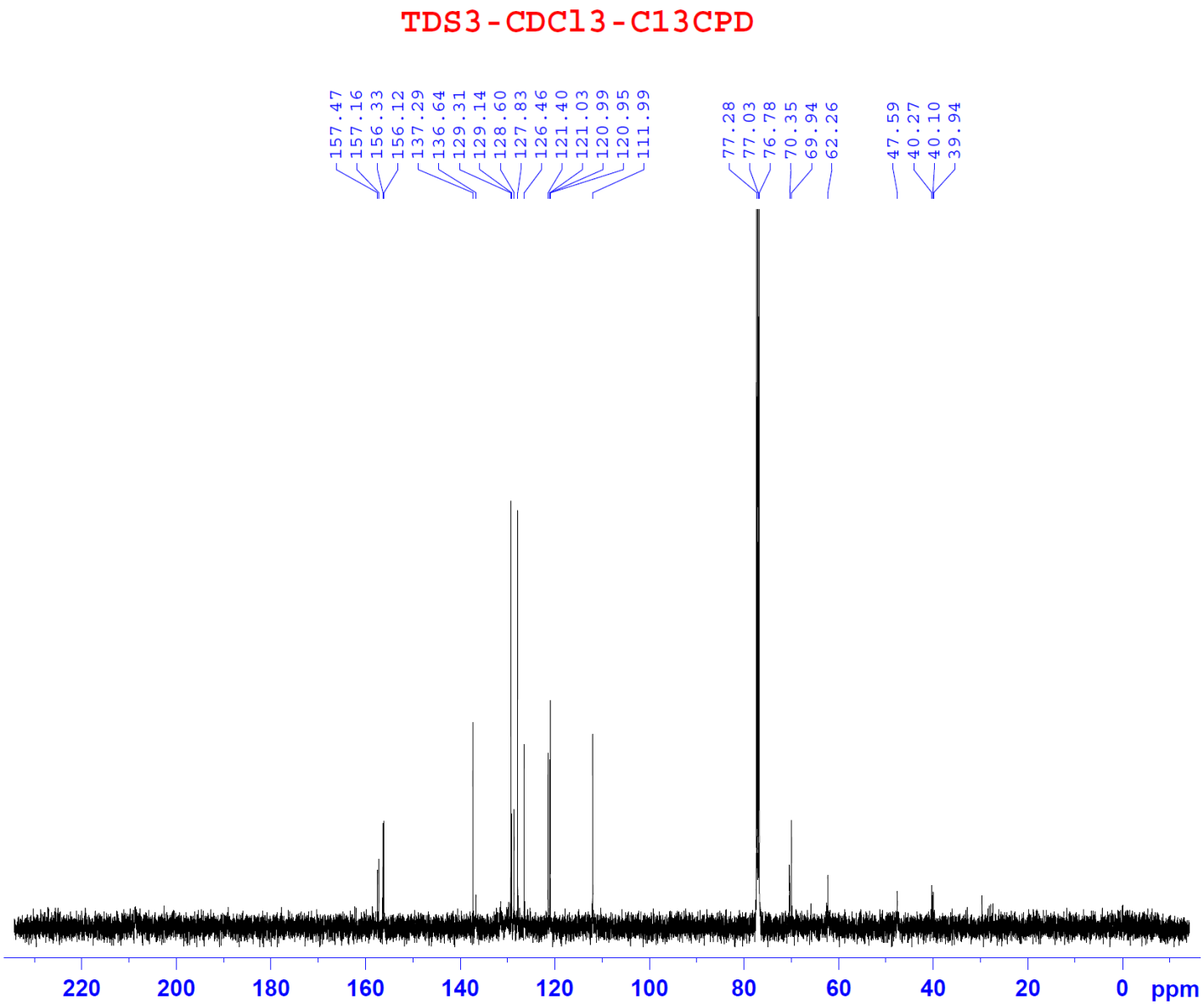
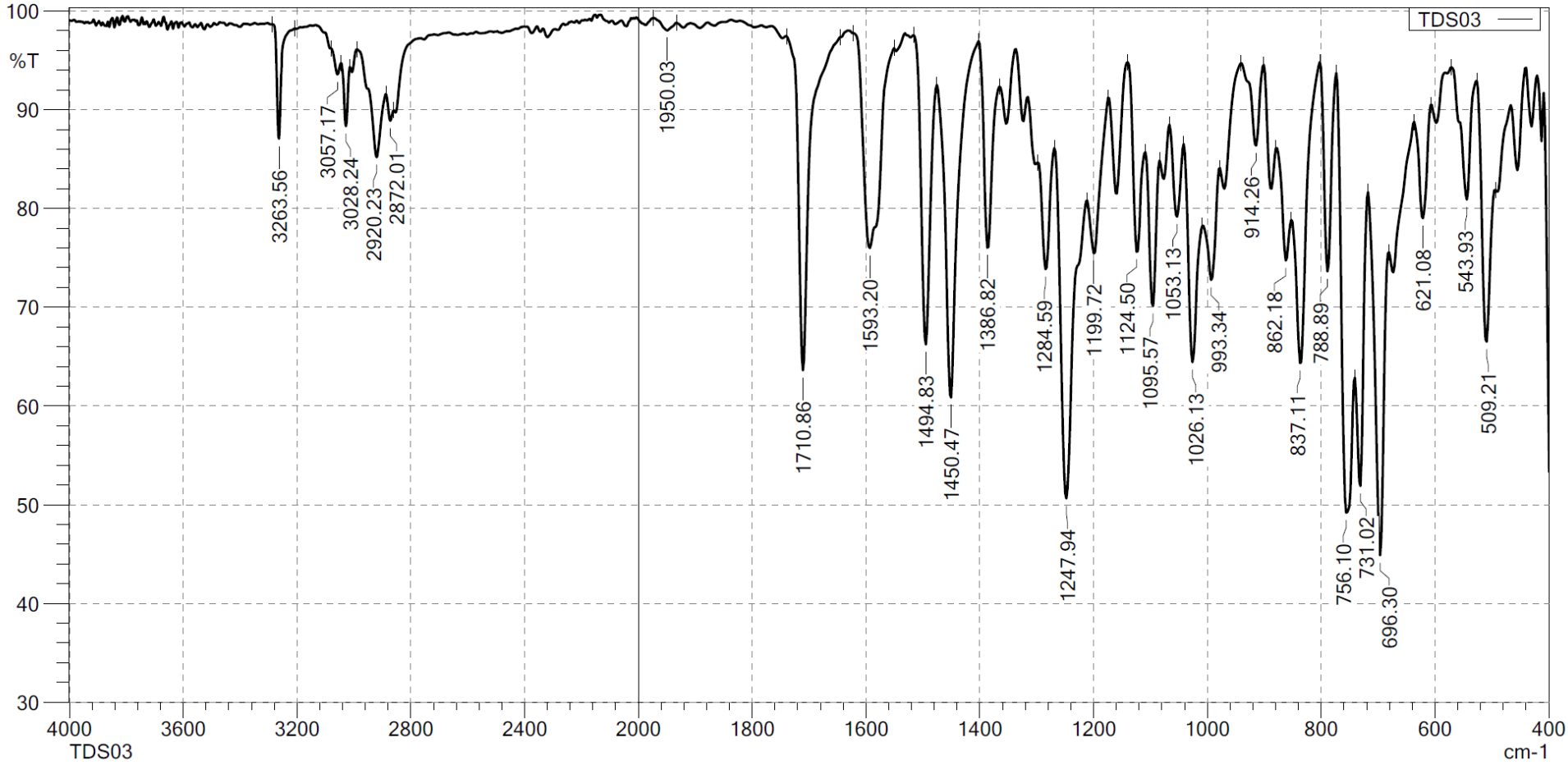
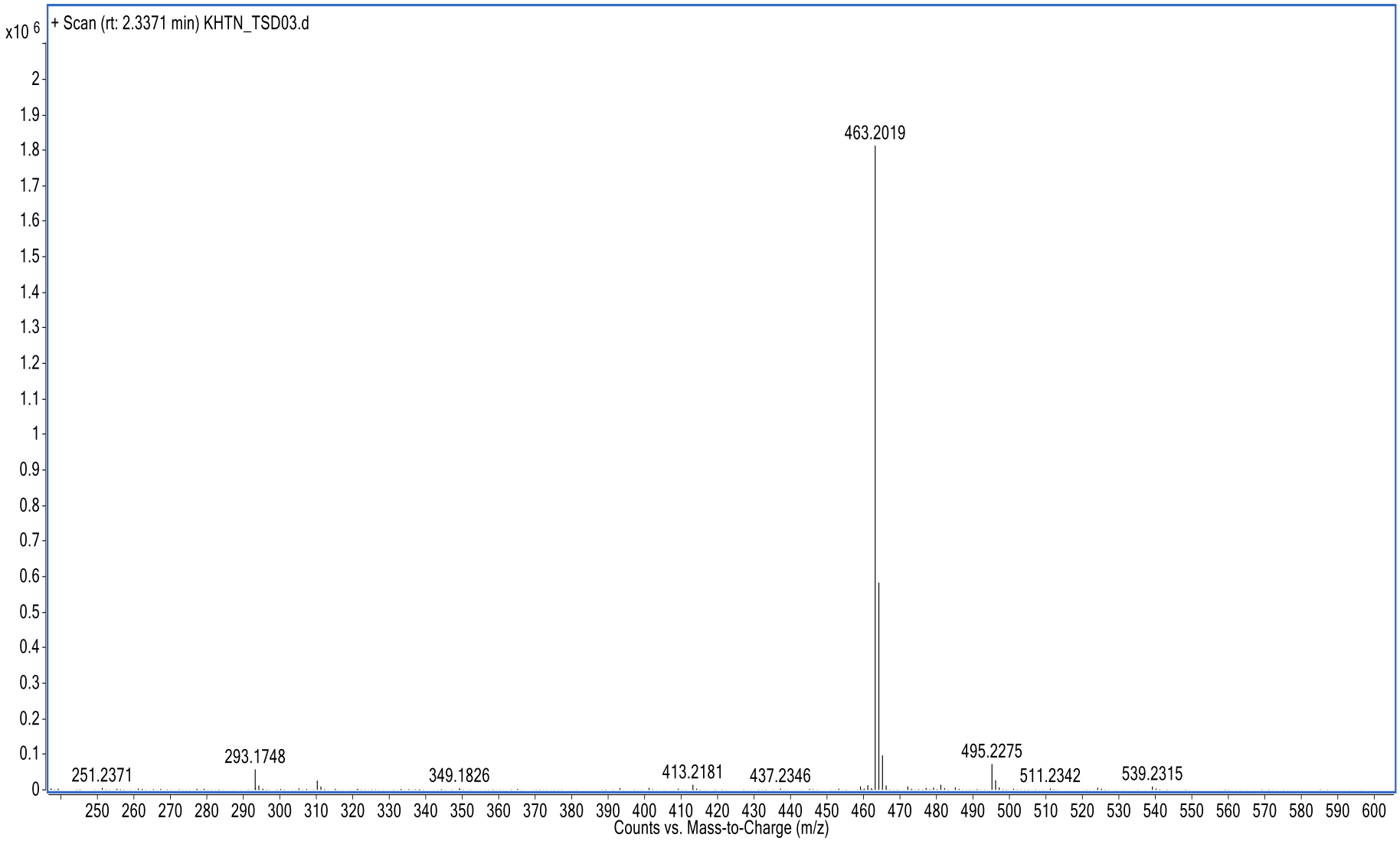
Picture S5. *^{1}H NMR spectrum of* ***2b***



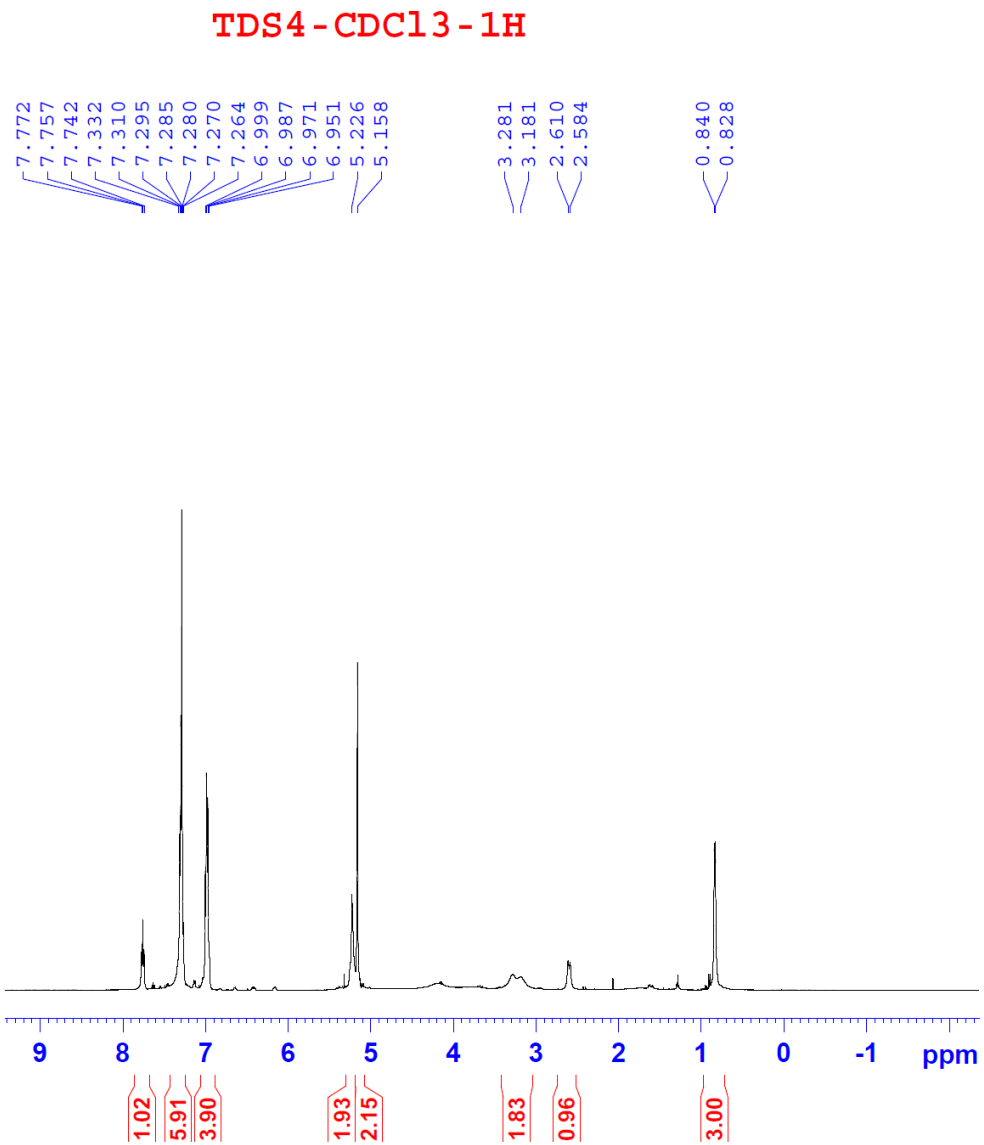
Picture S6. *^{13}C NMR spectrum of compound **2b***



Picture S7. *IR spectrum of compound* ***2b***



Picture S8. *HRMS spectrum of compound **2b***

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PROCNO                 1

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PULPROG             zg30
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NS                    16
DS                     2
SWH            10000.000 Hz
FIDRES          0.152588 Hz
AQ             3.2767999 sec
RG                175.34
DW                50.000 usec
DE                 6.50 usec
TE                302.4 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
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NUC1                  1H
P1                 7.90 usec
PLW1        13.19999981 W

F2 - Processing parameters
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SSB       0
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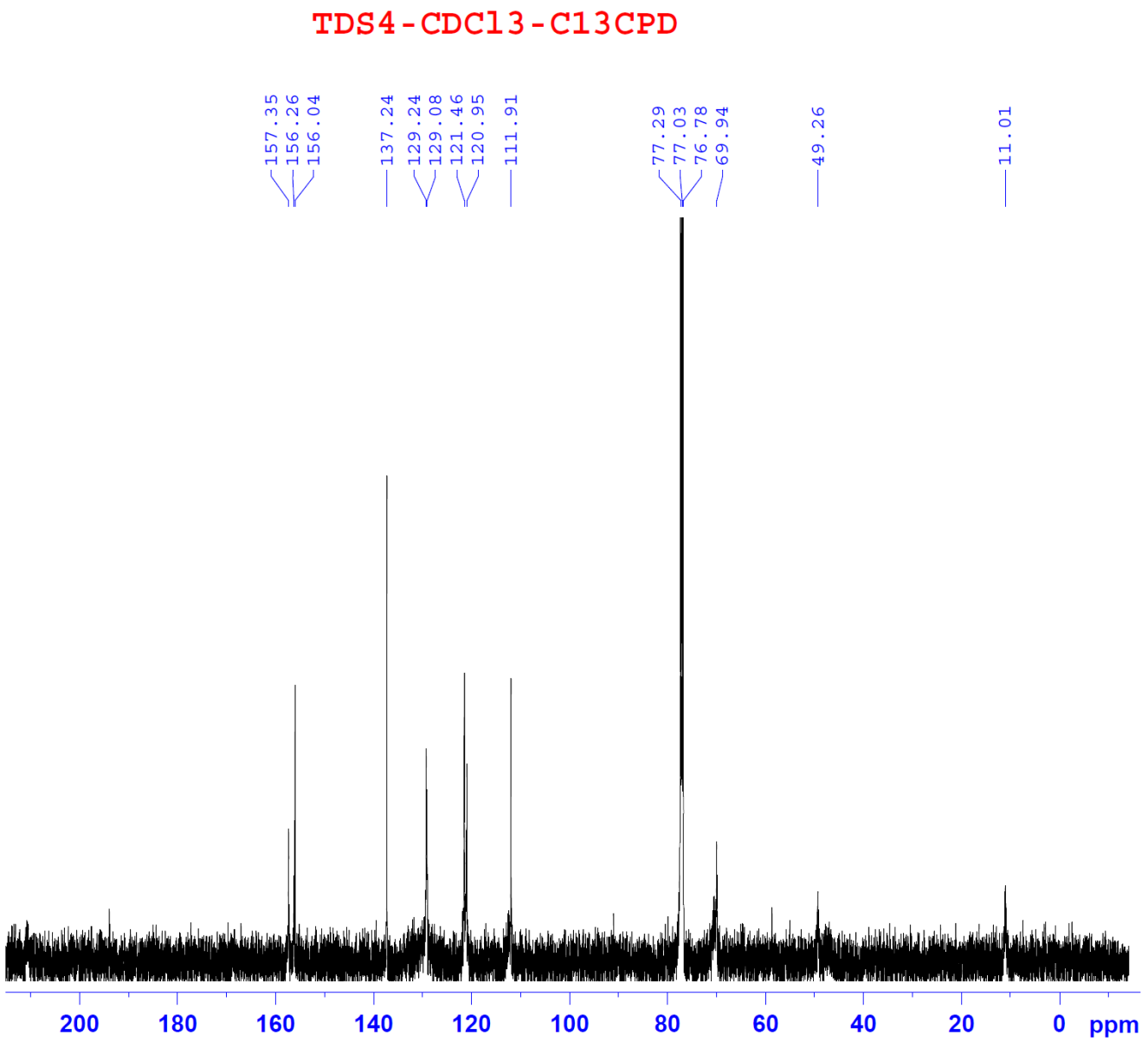
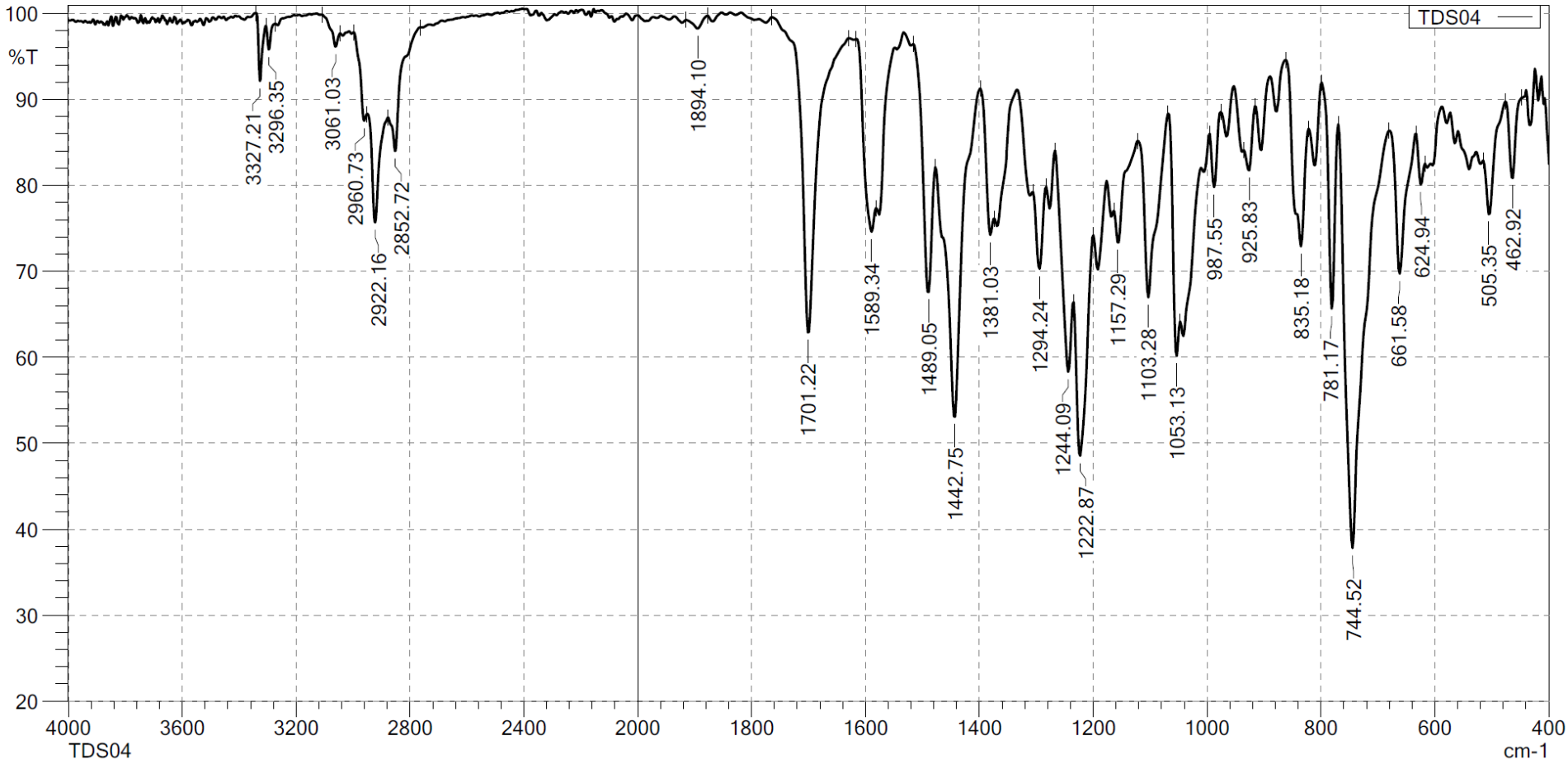
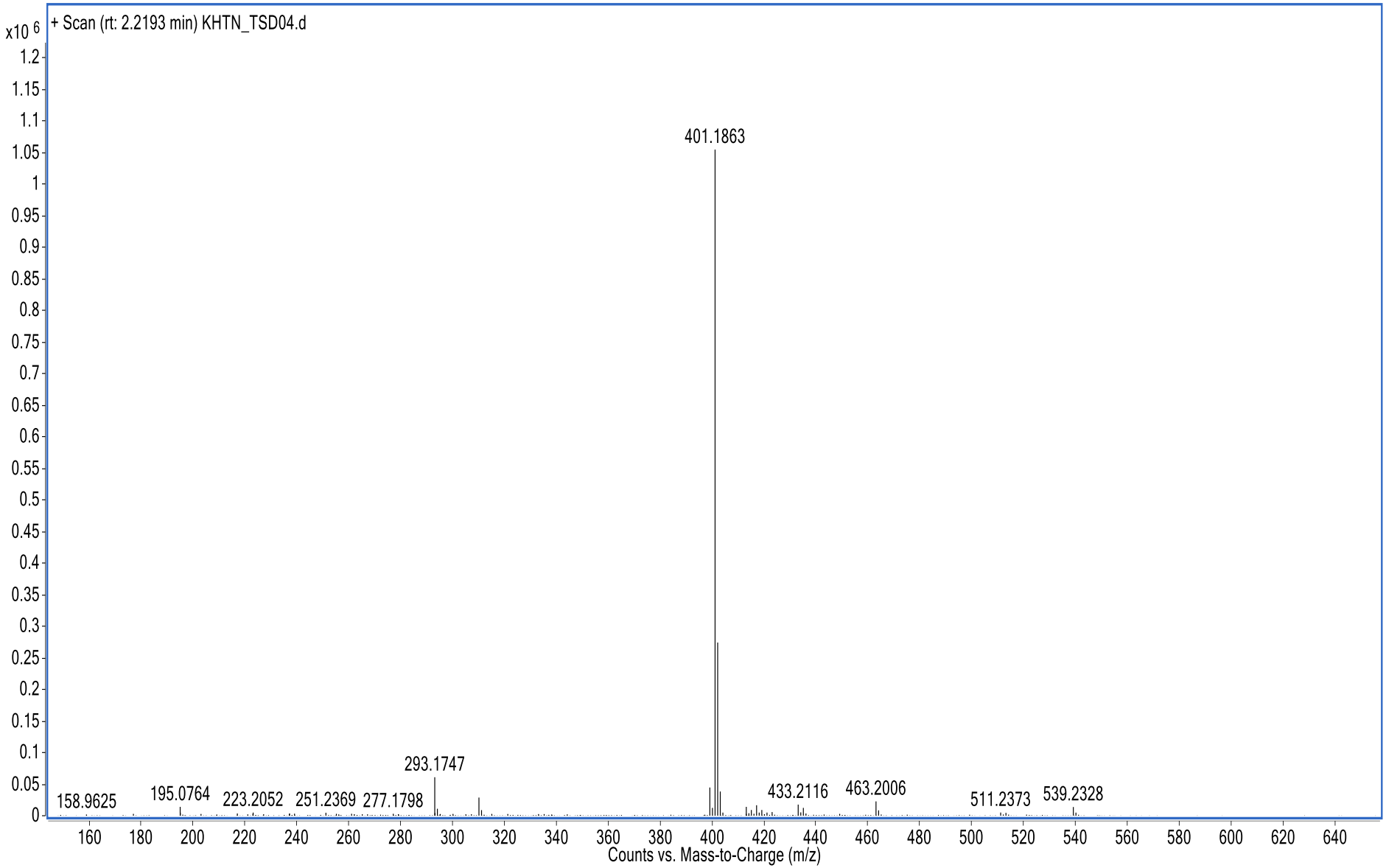
Picture S9. *^{1}H NMR spectrum of compound* ***2c***



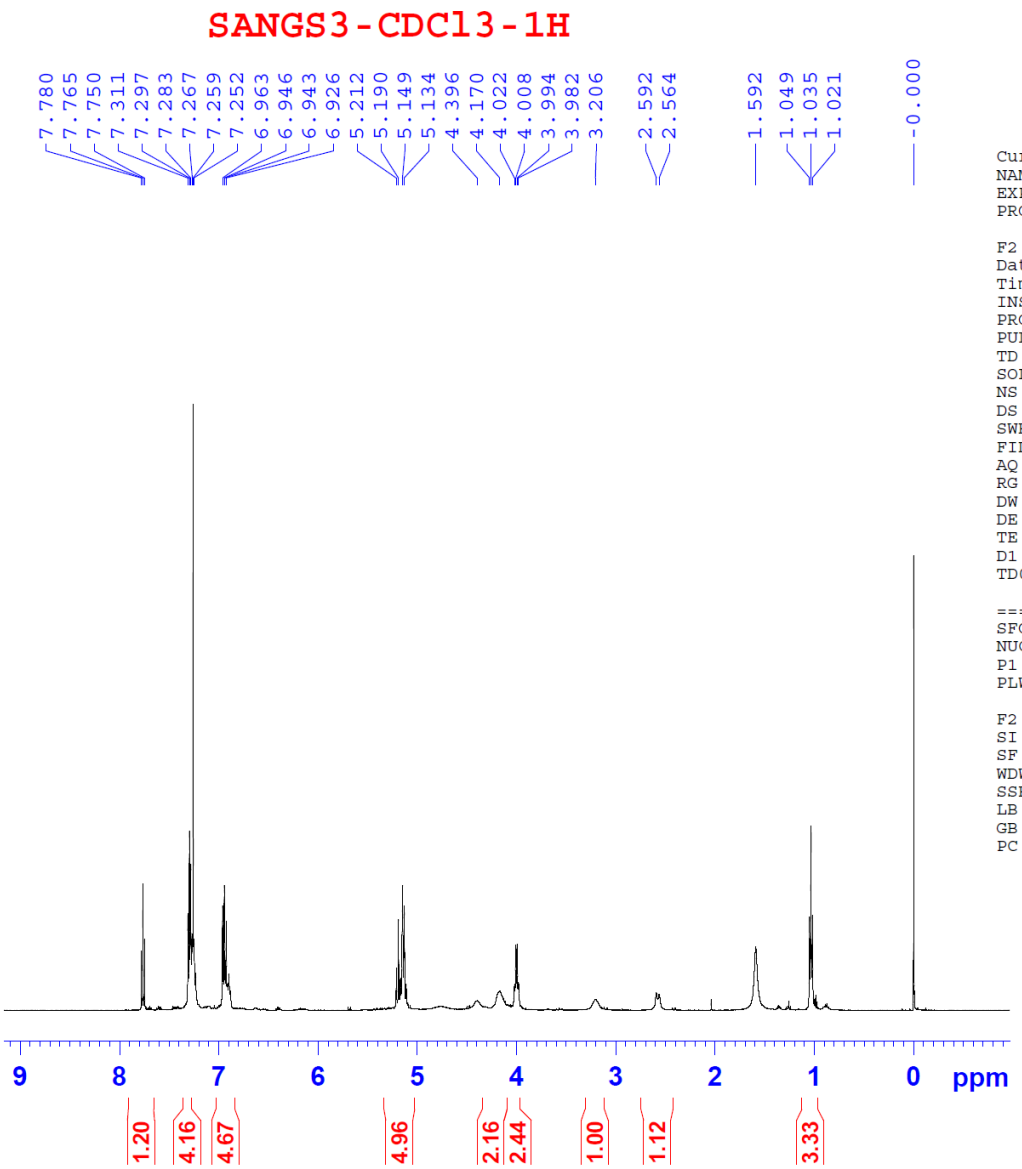
Picture S10. *^{13}C NMR spectrum compound **2c***



Picture S11. *IR spectrum of compound* ***2c***



Picture S12. *HRMS spectrum of compound* ***2c***

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NS                   16
DS                    2
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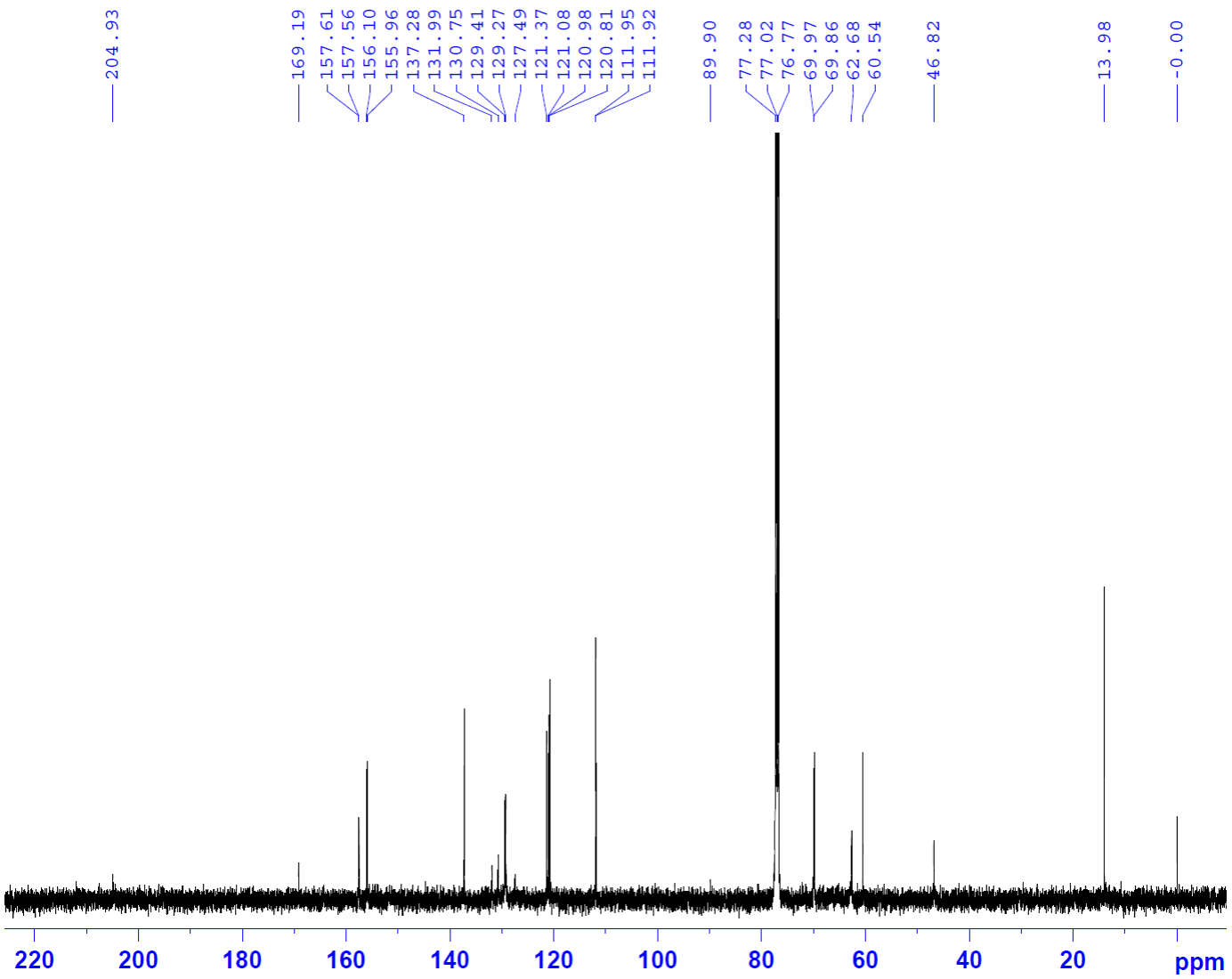
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG               175.34
DW               50.000 usec
DE                6.50 usec
TE               303.0 K
D1           1.00000000 sec
TD0                   1

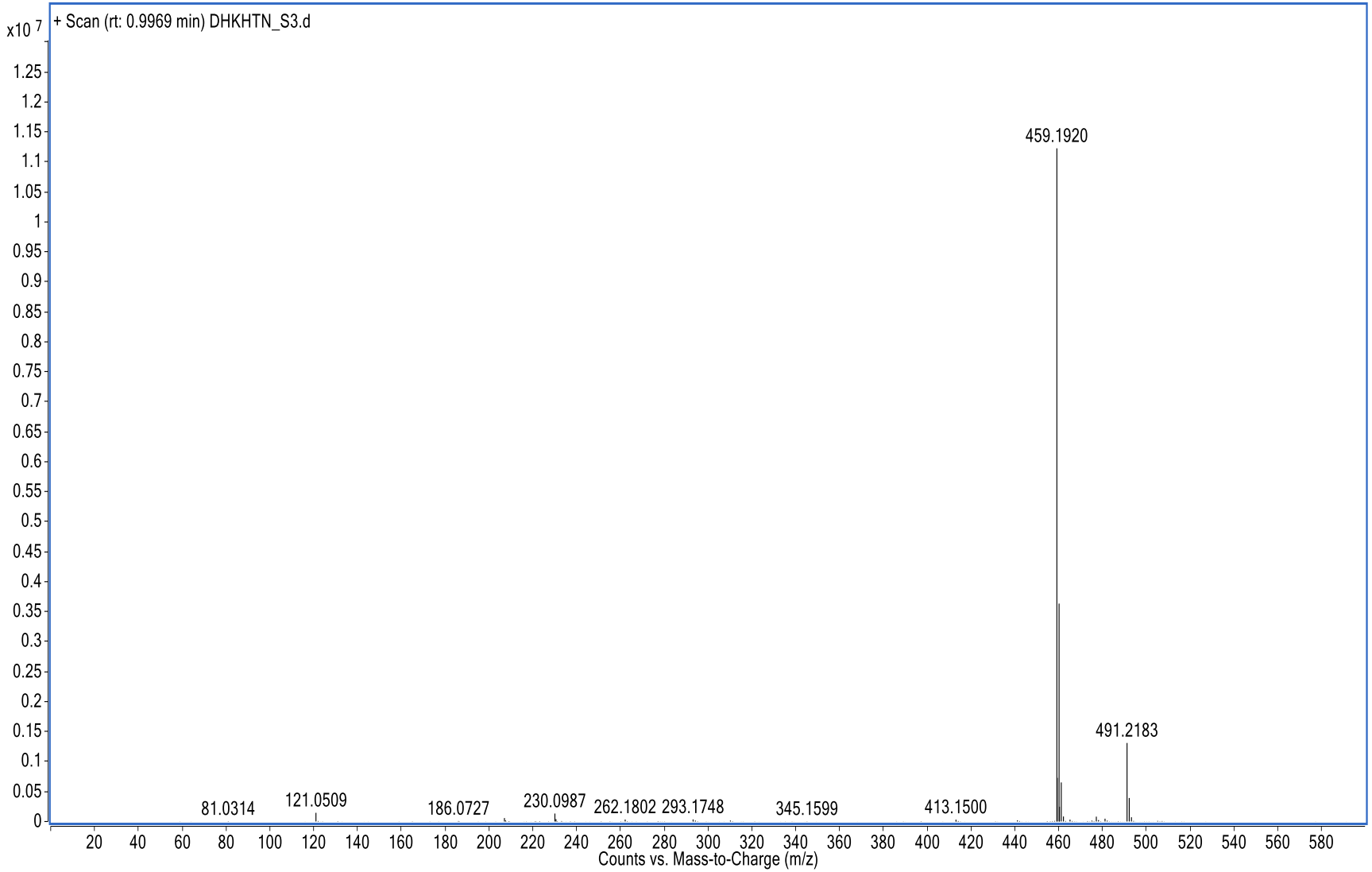
======== CHANNEL f1 ========
SFO1         500.2049890 MHz
NUC1                 1H
P1                10.00 usec
PLW1         22.00000000 W

F2 - Processing parameters
SI                65536
SF           500.2019131 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

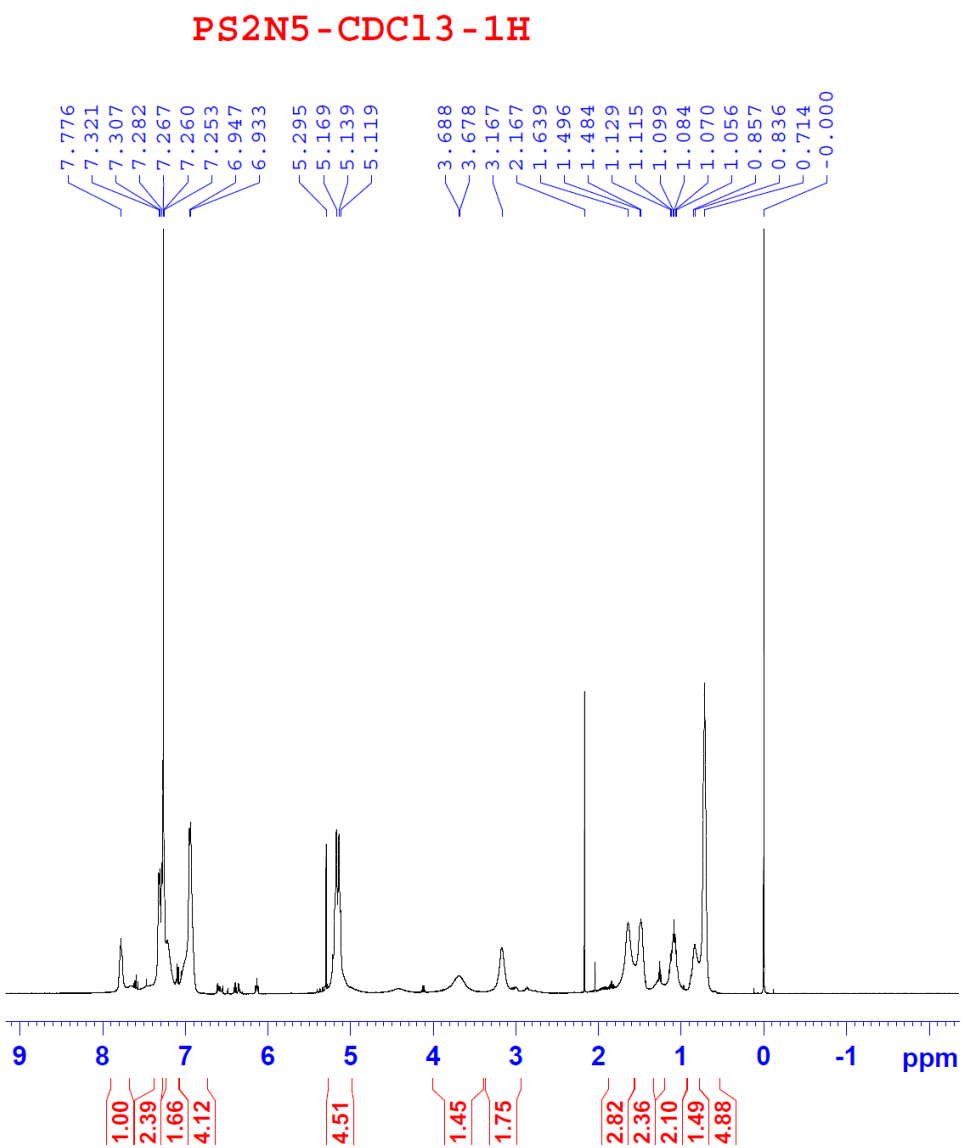
Picture S13. *^{1}H NMR Spectrum of* ***2d***



Picture S14. *^{13}C NMR Spectrum of* ***2d***



Picture S15. *HRMS spectrum of compound* ***2d***



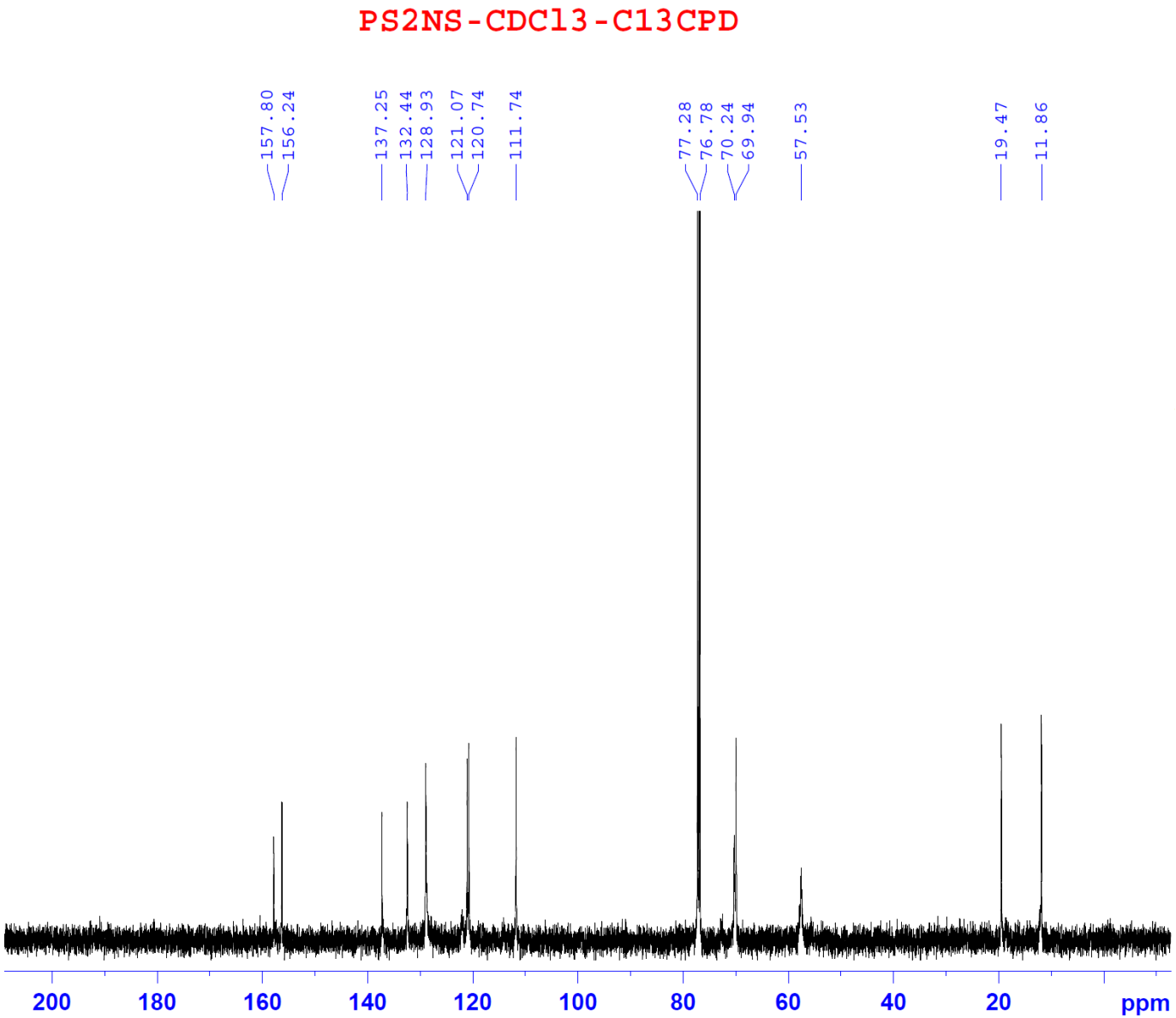

```
Current Data Parameters
NAME                  DAT_PS2N5
EXPNO                        10
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20190830
Time                     16.35
INSTRUM                  spect
PROBHD    5 mm PABBI 1H/
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                  10000.000 Hz
FIDRES                0.152588 Hz
AQ                    3.2767999 sec
RG                       198.57
DW                       50.000 usec
DE                        6.50 usec
TE                       302.5 K
D1                   1.00000000 sec
TD0                          1

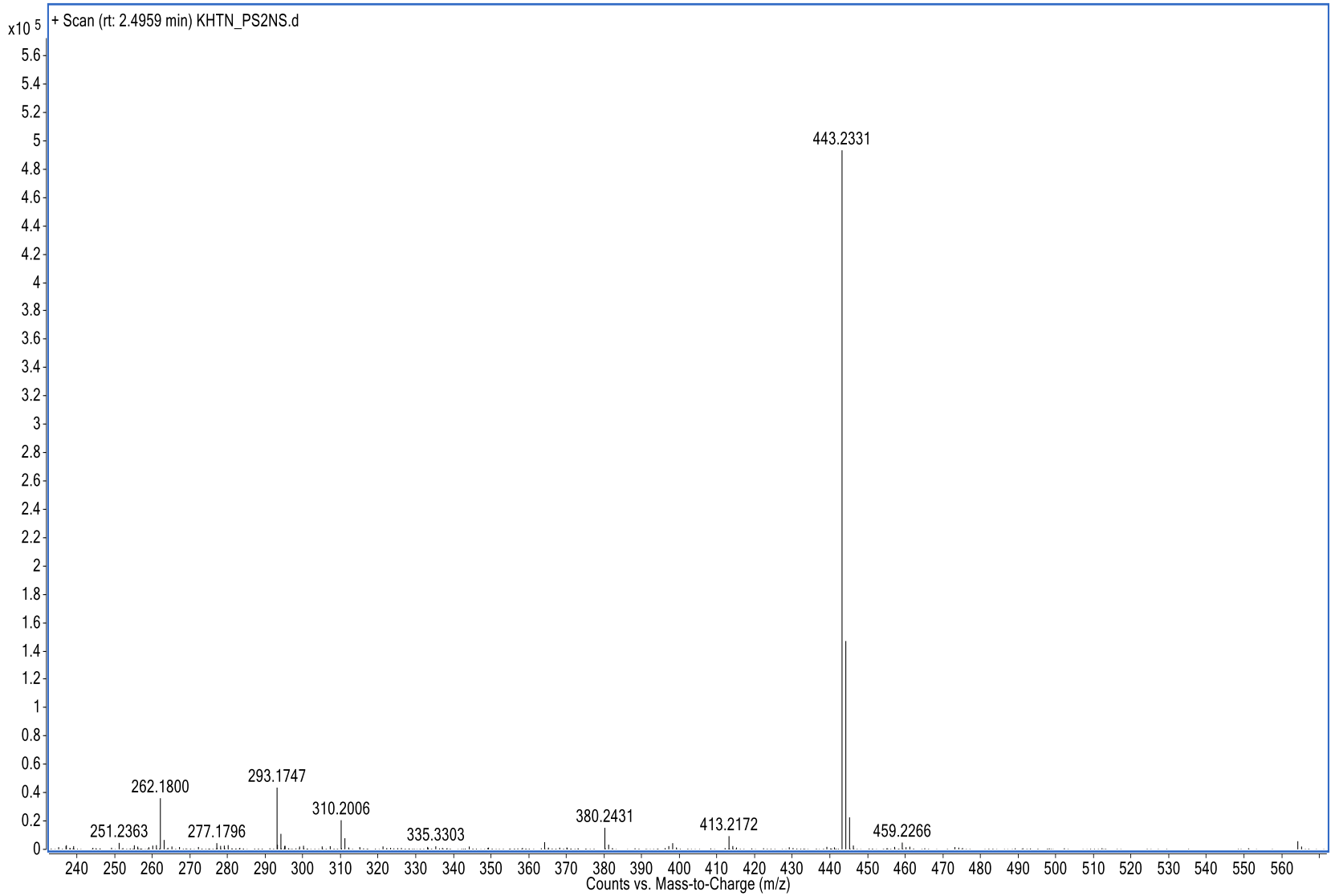
======== CHANNEL f1 ========
SFO1                500.1920889 MHz
NUC1                        1H
P1                        7.90 usec
PLW1                13.19999981 W

F2 - Processing parameters
SI                       65536
SF                  500.1890128 MHz
WDW                         EM
SSB                  0
LB                        0.30 Hz
GB                   0
PC                        1.00
```

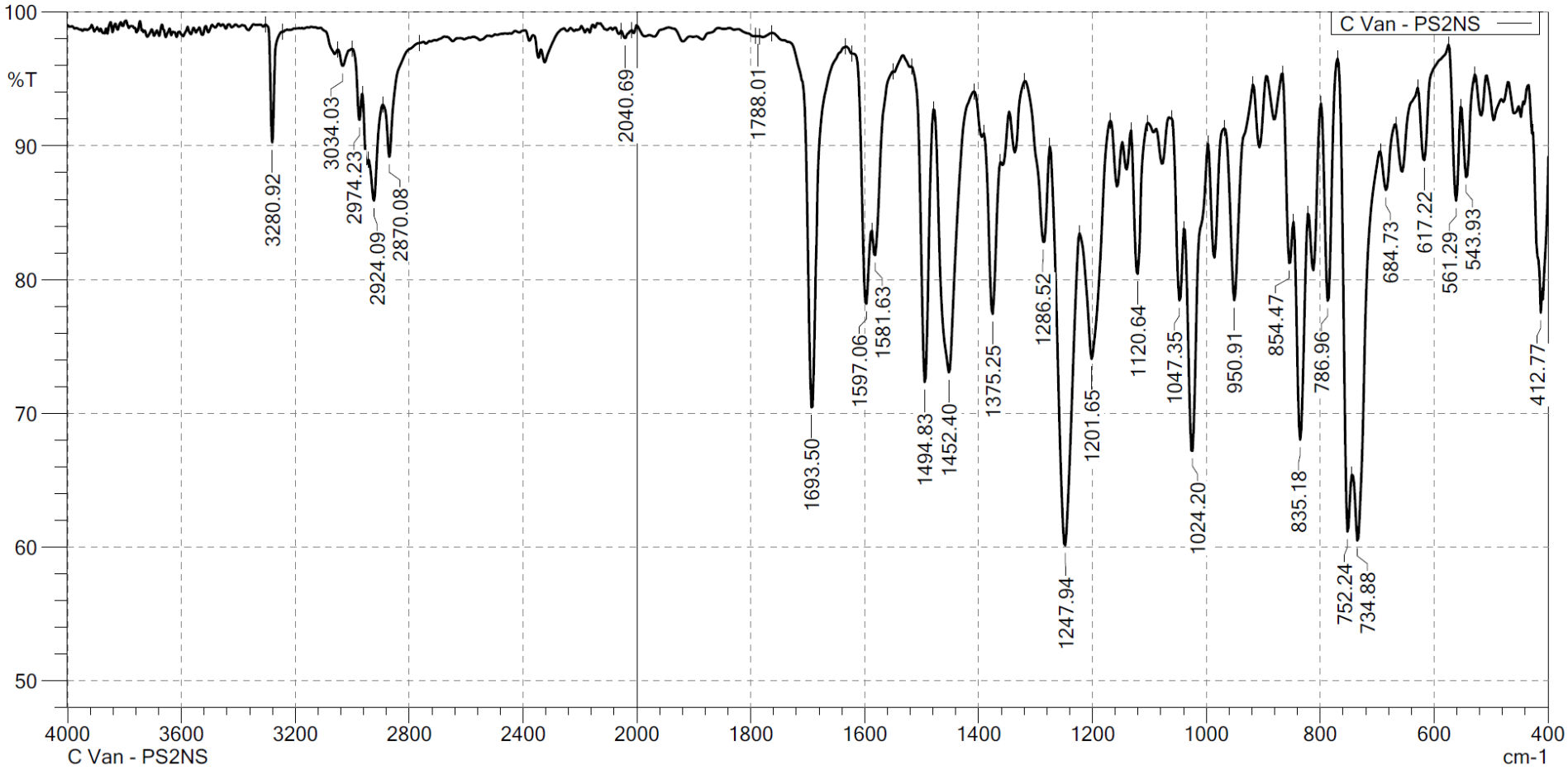
Picture S16. *^{1}H NMR spectrum of* ***2e***



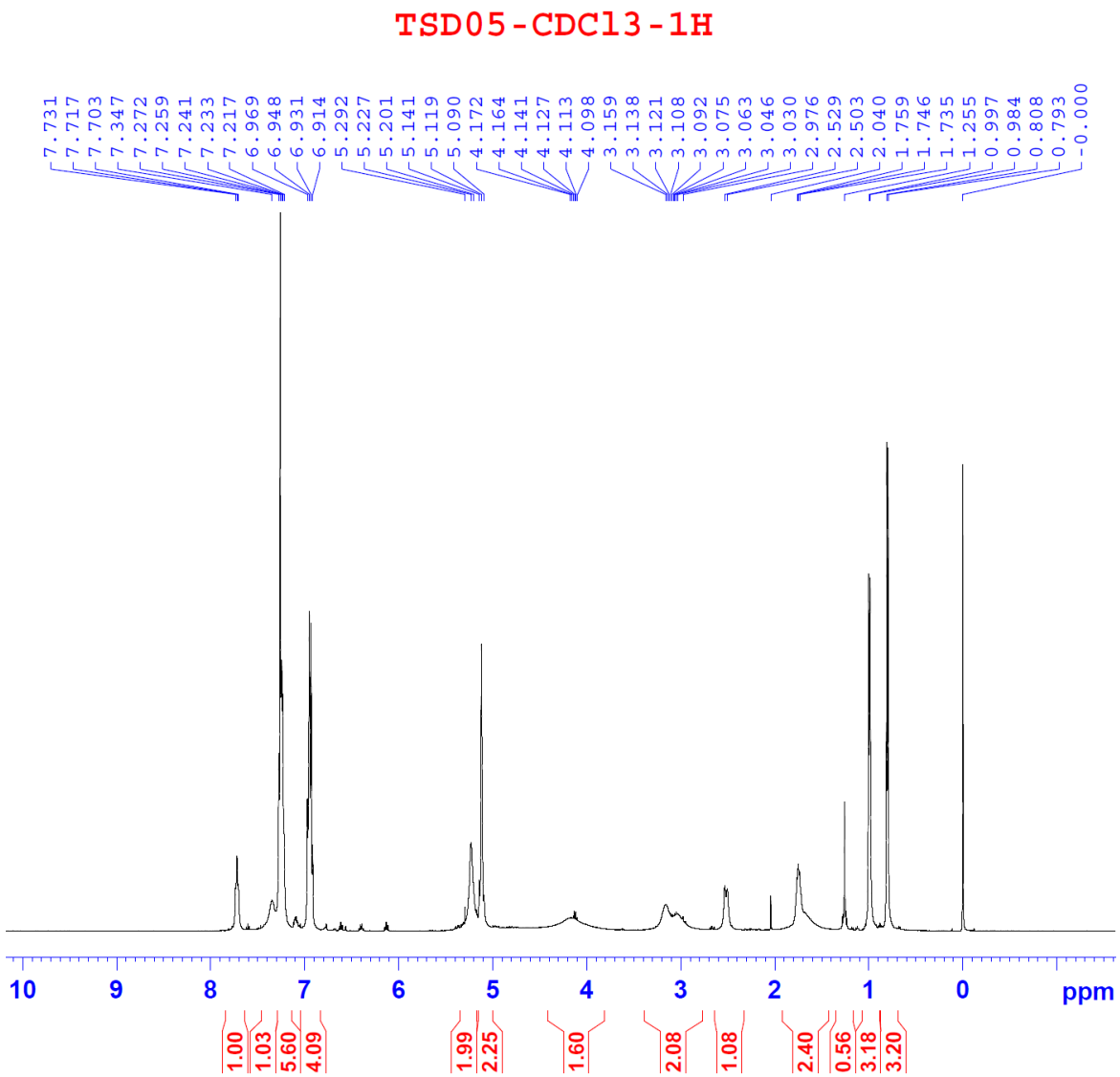
Picture S17. *^{13}C NMR of compound **2e***



Picture S18. *HRMS Spectrum of compound* ***2e***



Picture S19. *IR Spectrum of compound* ***2e***



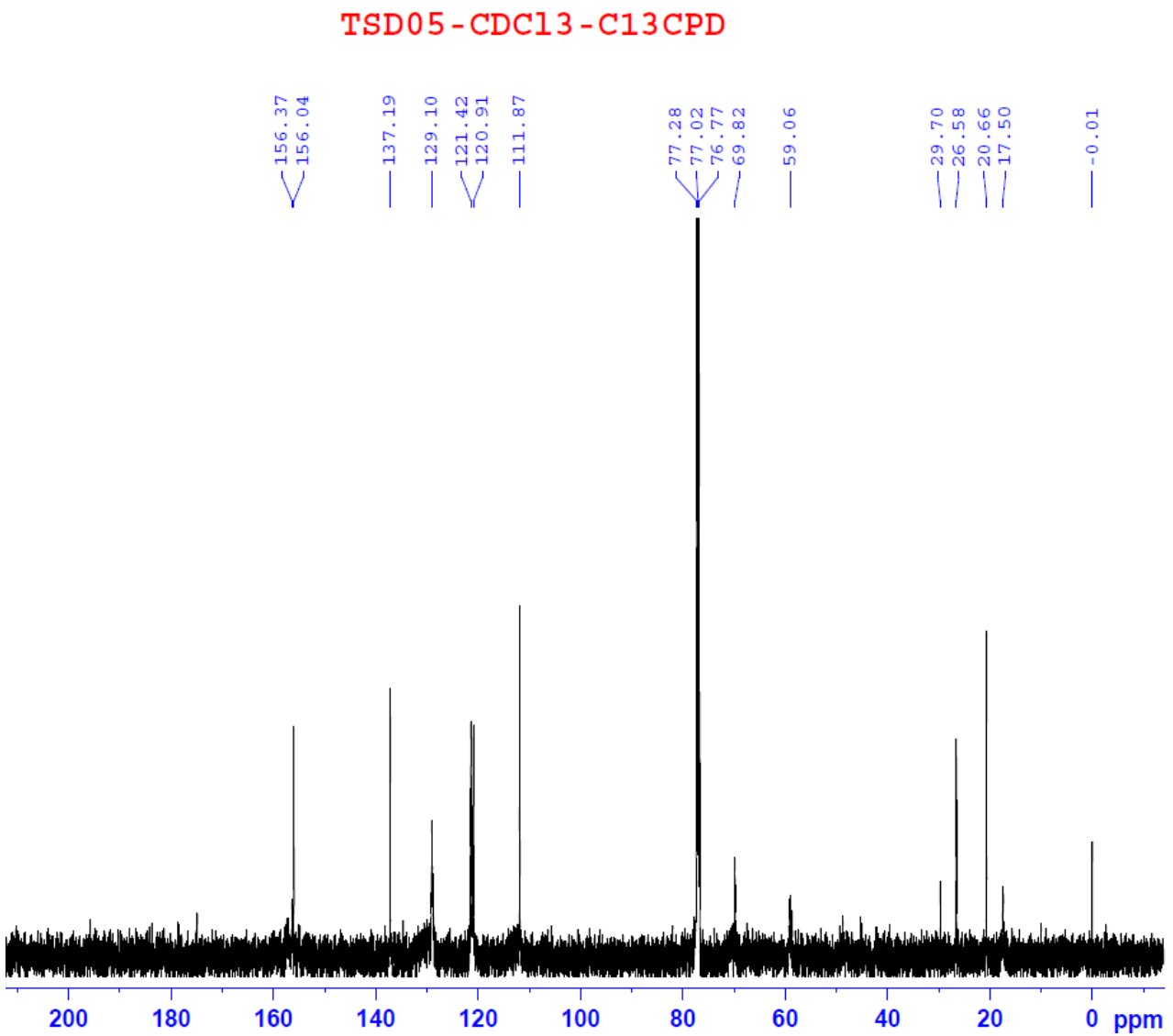

```
Current Data Parameters
NAME            DAT_TSD05
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20190906
Time                12.55
INSTRUM             spect
PROBHD    5 mm PABBI 1H/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH             10000.000 Hz
FIDRES              0.152588 Hz
AQ              3.2767999 sec
RG                 157.35
DW                 50.000 usec
DE                  6.50 usec
TE                 302.6 K
D1             1.00000000 sec
TD0                     1

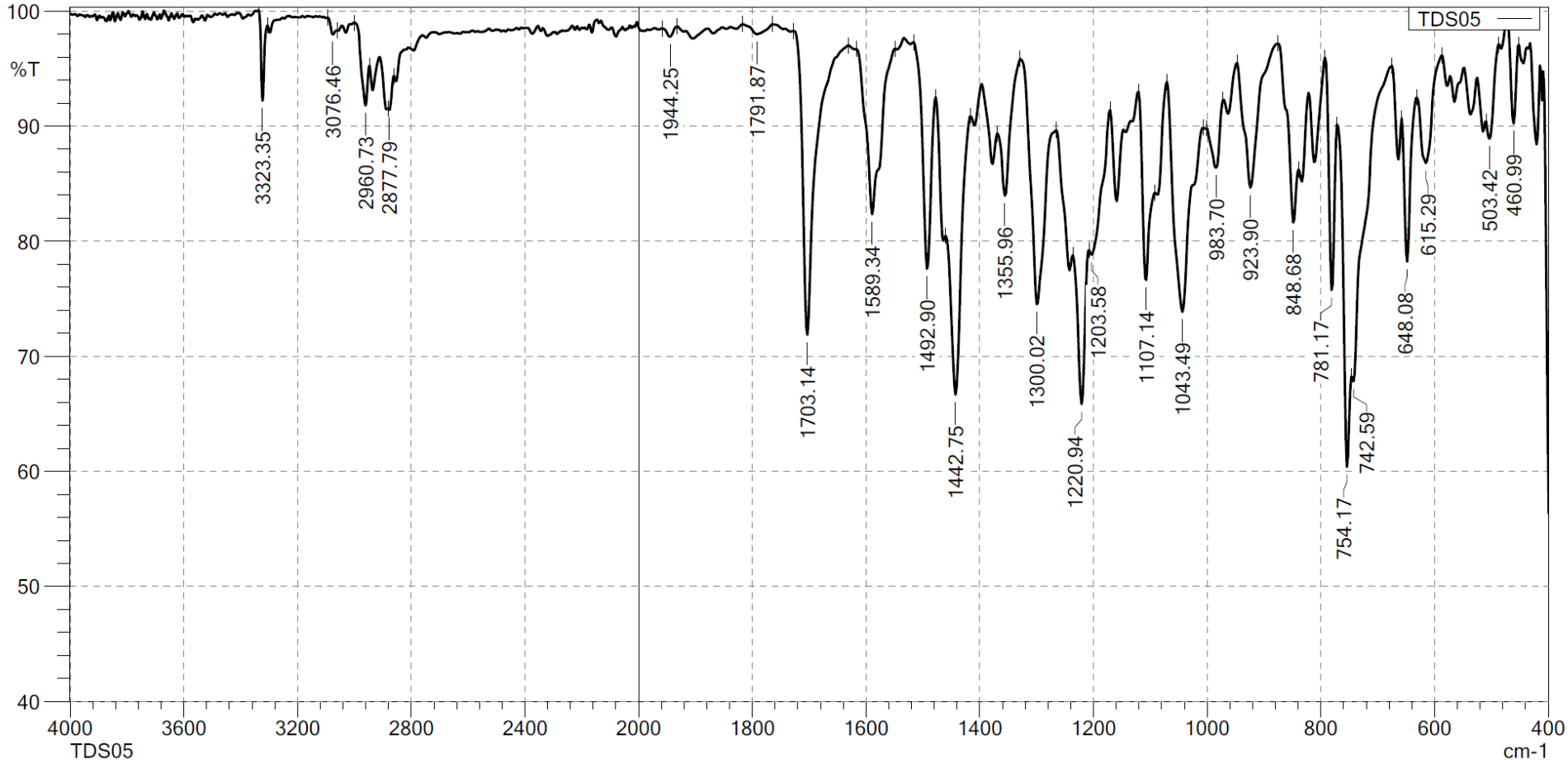
======== CHANNEL f1 ========
SFO1          500.1920889 MHz
NUC1                   1H
P1                   7.90 usec
PLW1          13.19999981 W

F2 - Processing parameters
SI                  65536
SF            500.1890136 MHz
WDW                    EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```

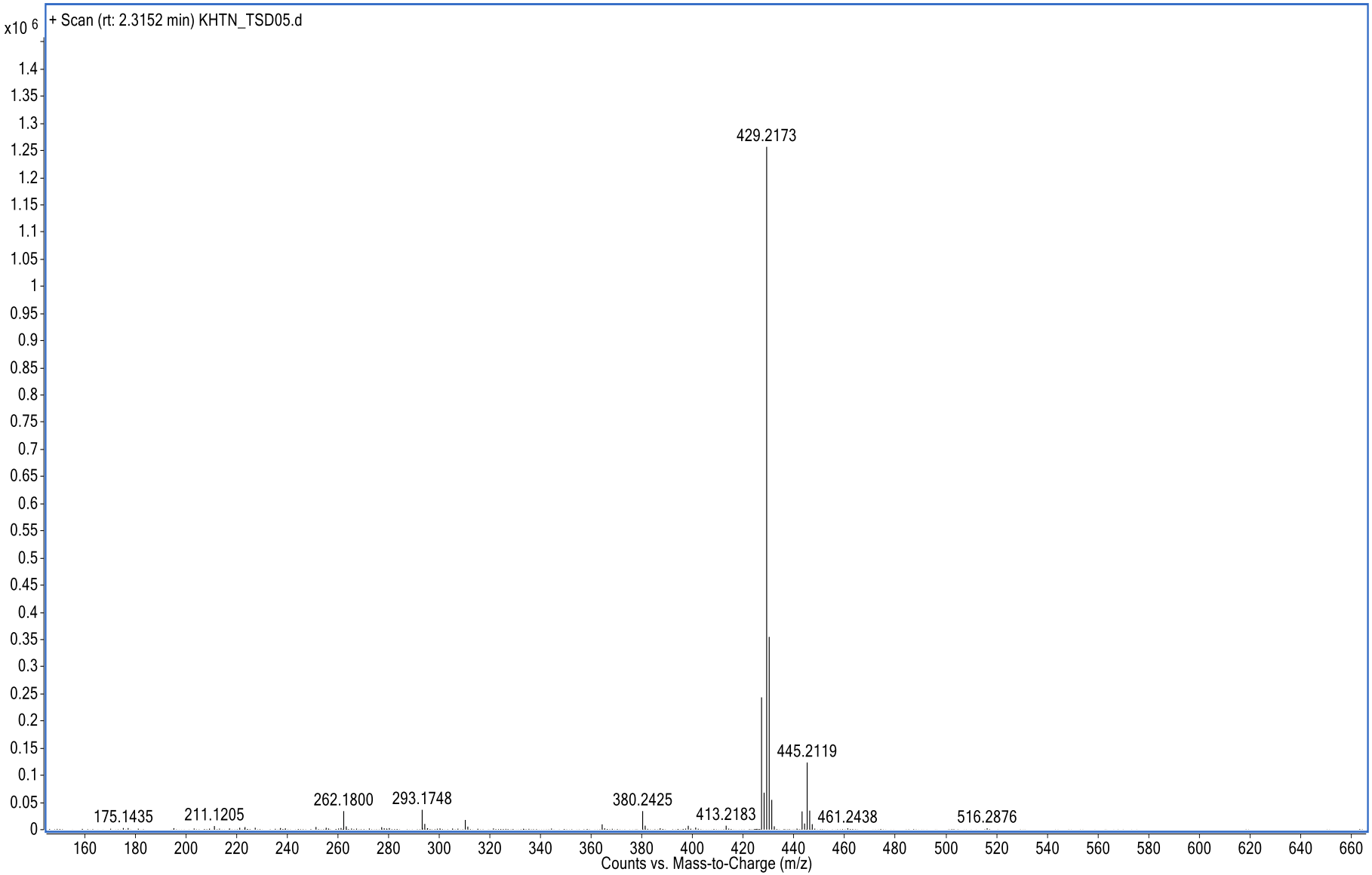
Picture S20. *[1]H NMR spectrum of* ***2f***



Picture S21. *^{13}C NMR spectrum of **2f***



Picture S22. *IR Spectrum of compound **2f***



Picture S23. *HRMS spectrum of compound **2f***