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Supplementary Information

Phosphine-free Ru(II)-CNC pincer complexes with mixed protic- and classical-NHCs in the same molecule for hydrogen production via oxidant-free benzyl alcohol dehydrogenation to benzoic acids

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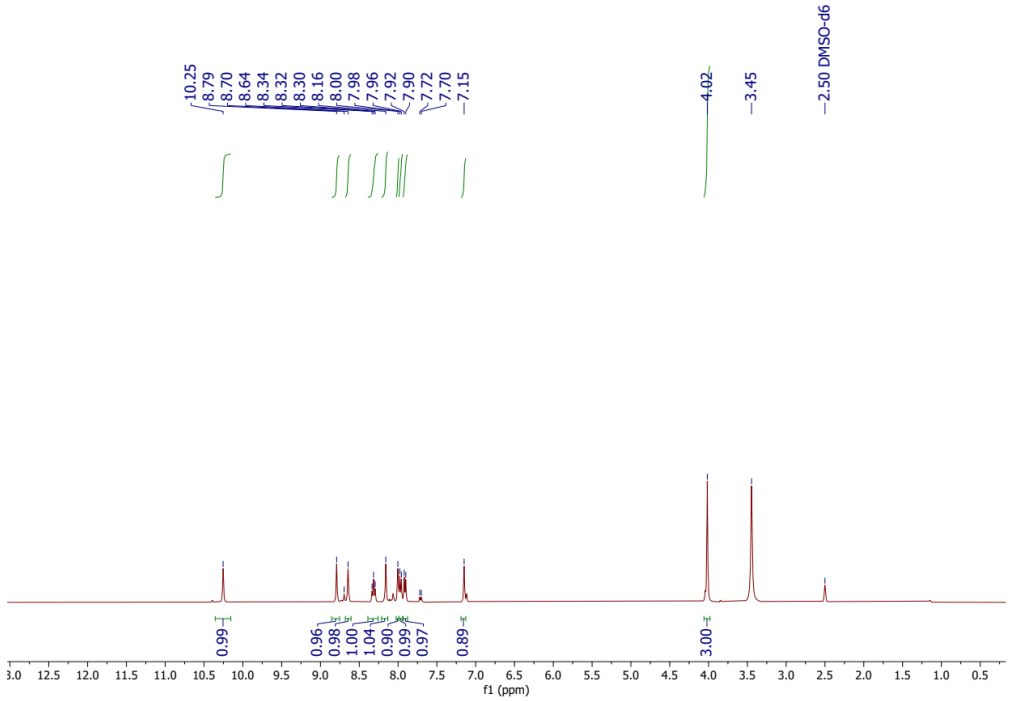


Figure S1. ^{1}H NMR spectrum of **L^{1}.HI.**

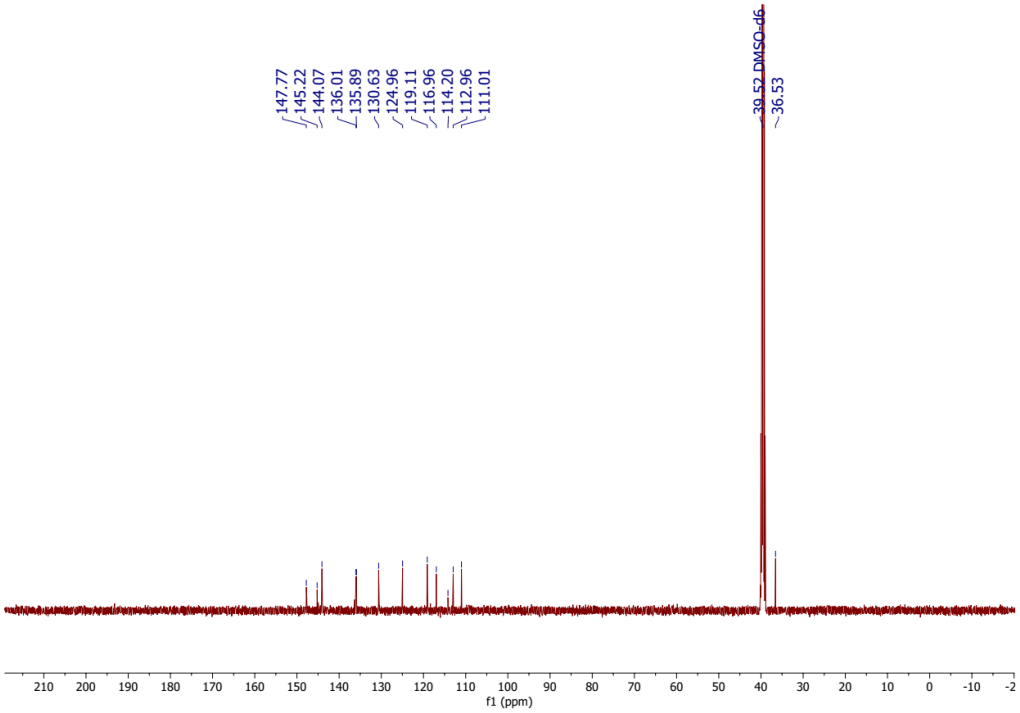


Figure S2. ^{13}C{^{1}H} NMR spectrum of **L^{1}·HI.**

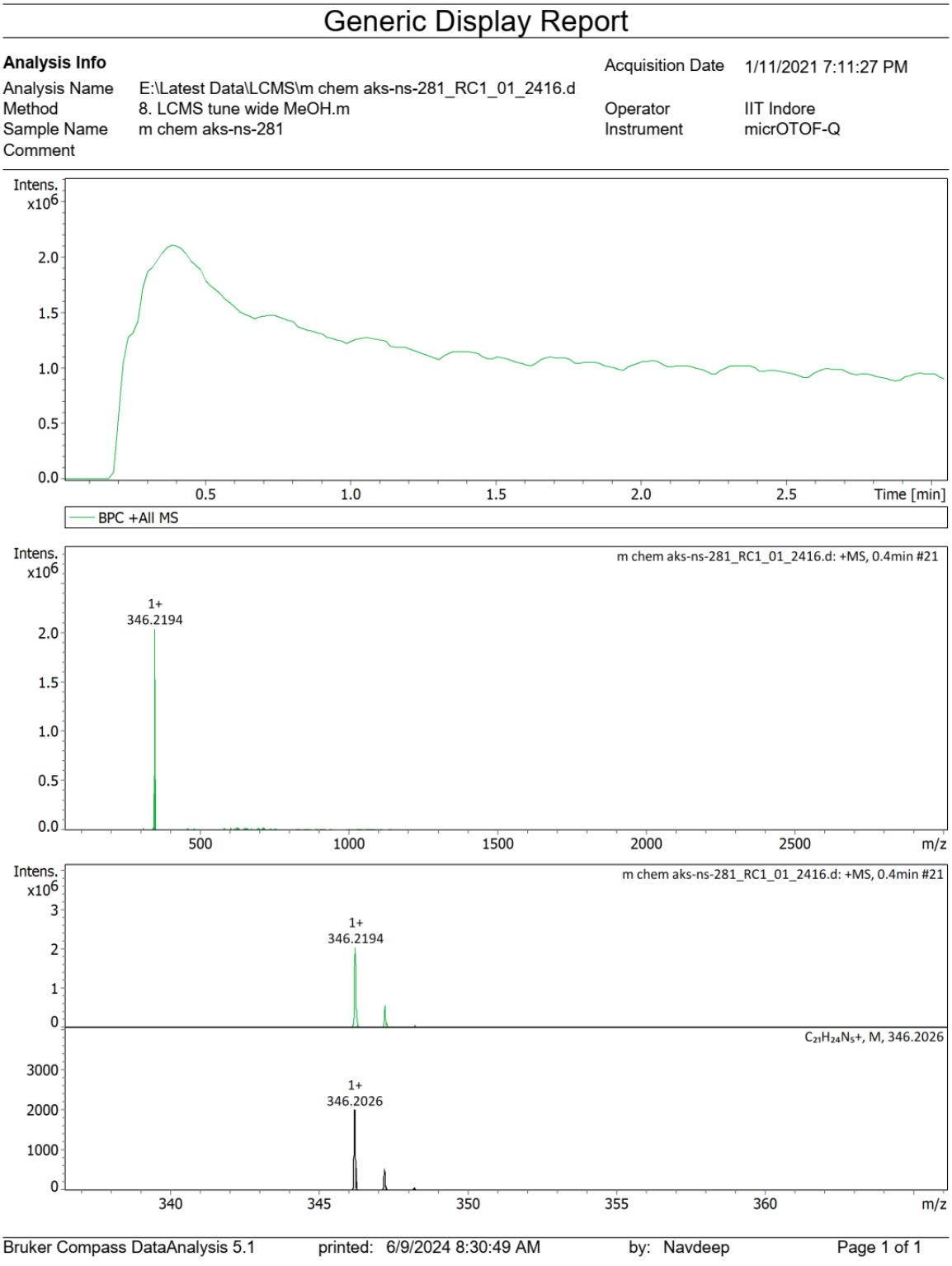


Figure S3 ESI-MS of L2·HBr ligand.

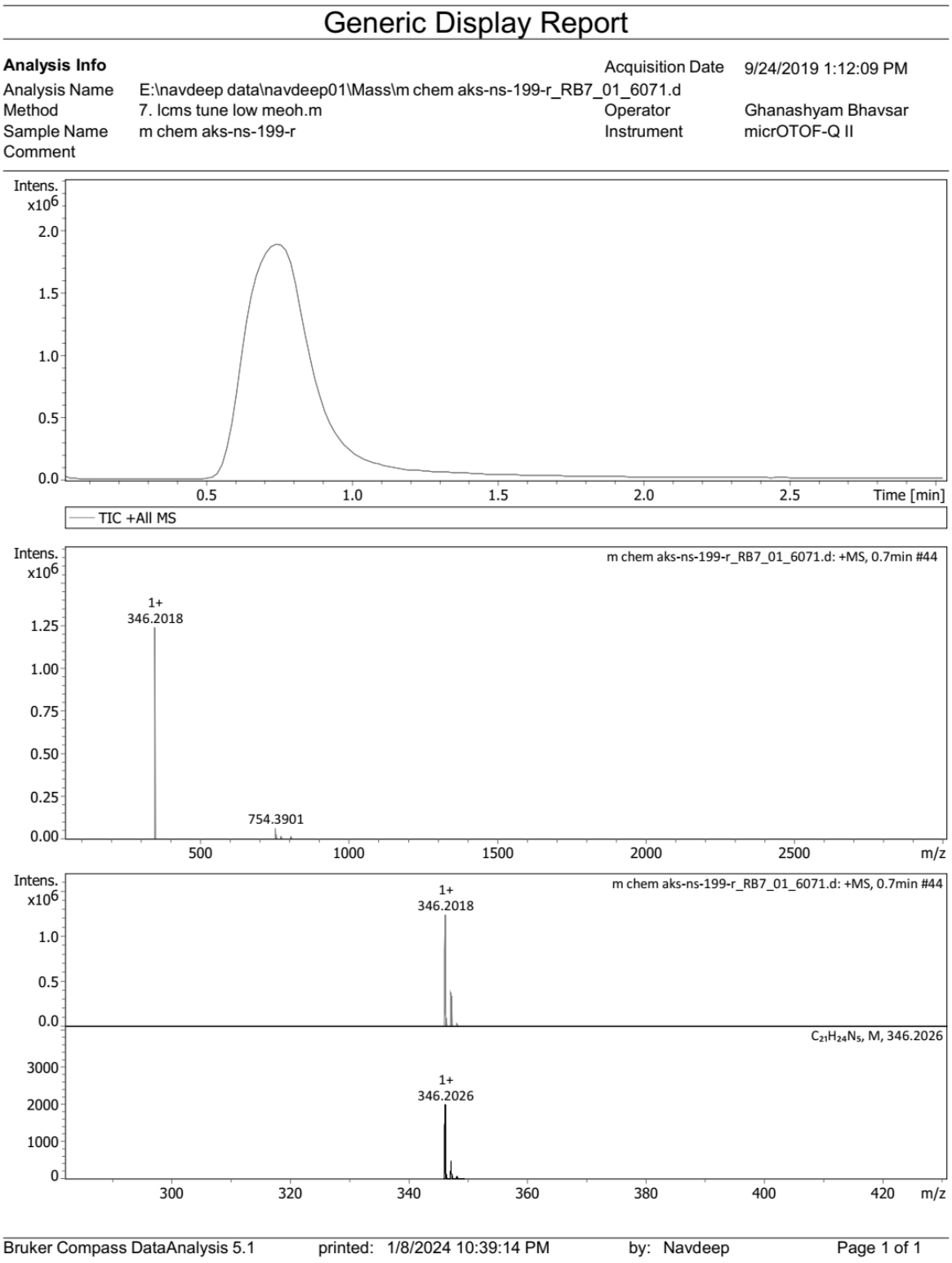


Figure S4. HRMS of L2·HBr ligand.

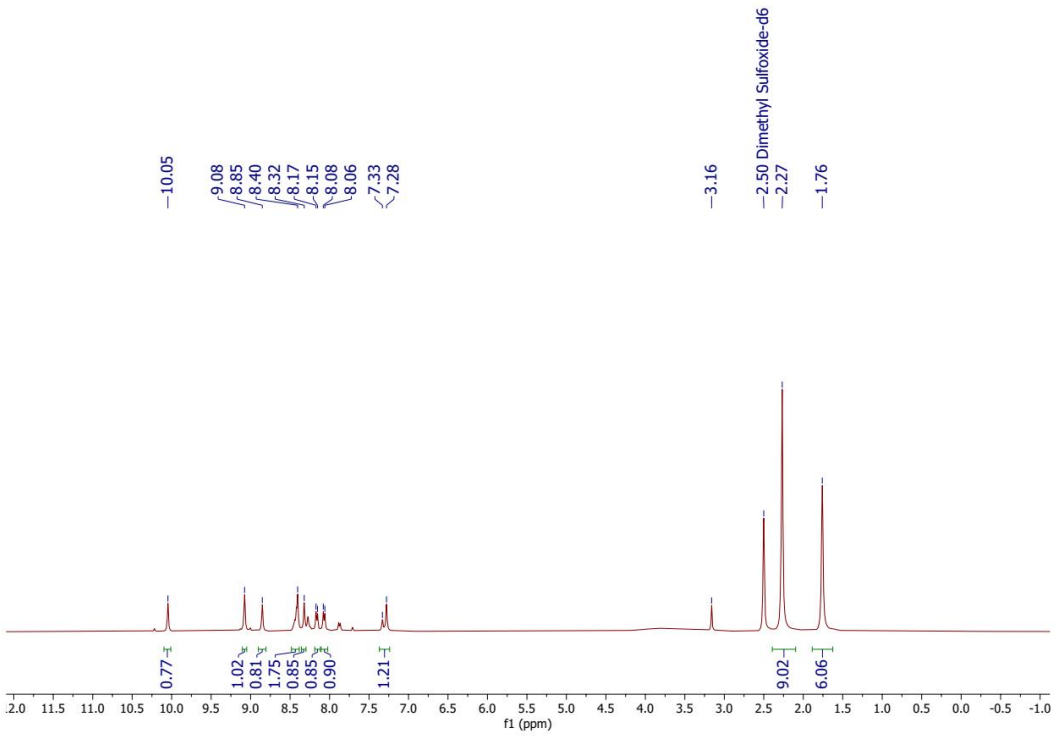


Figure S5. ^{1}H NMR of L2·HBr ligand.

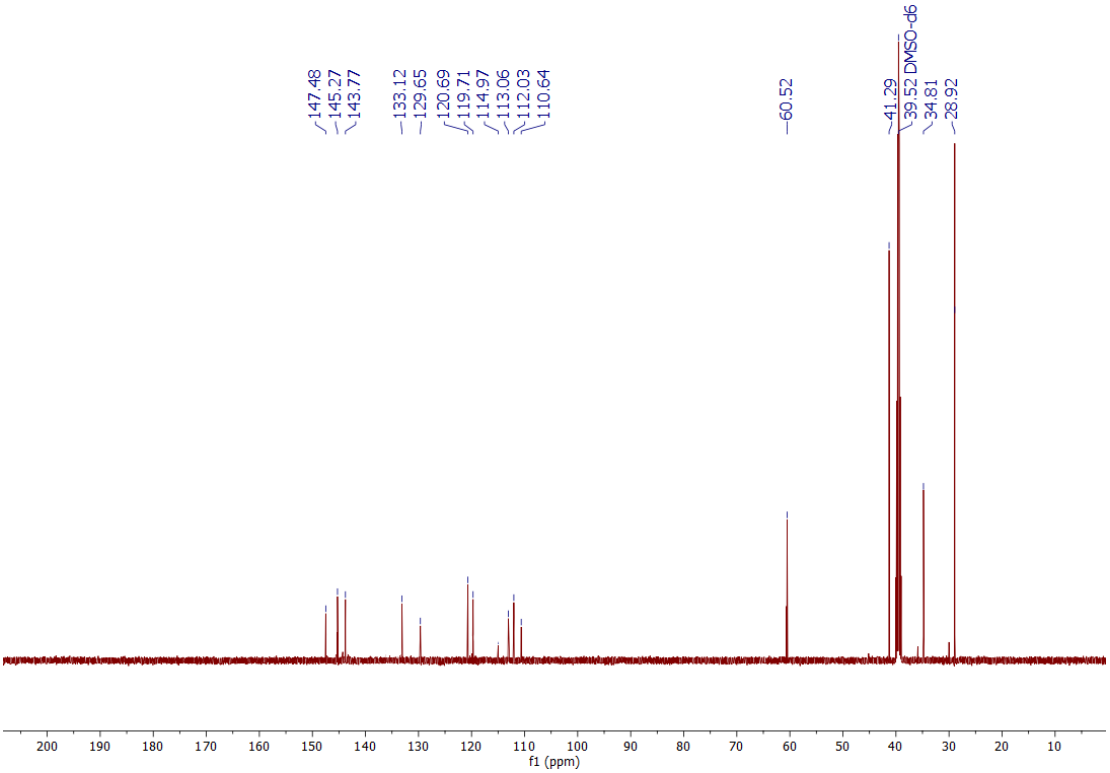


Figure S6. ^{13}C{^{1}H} NMR of L2·HBr ligand.

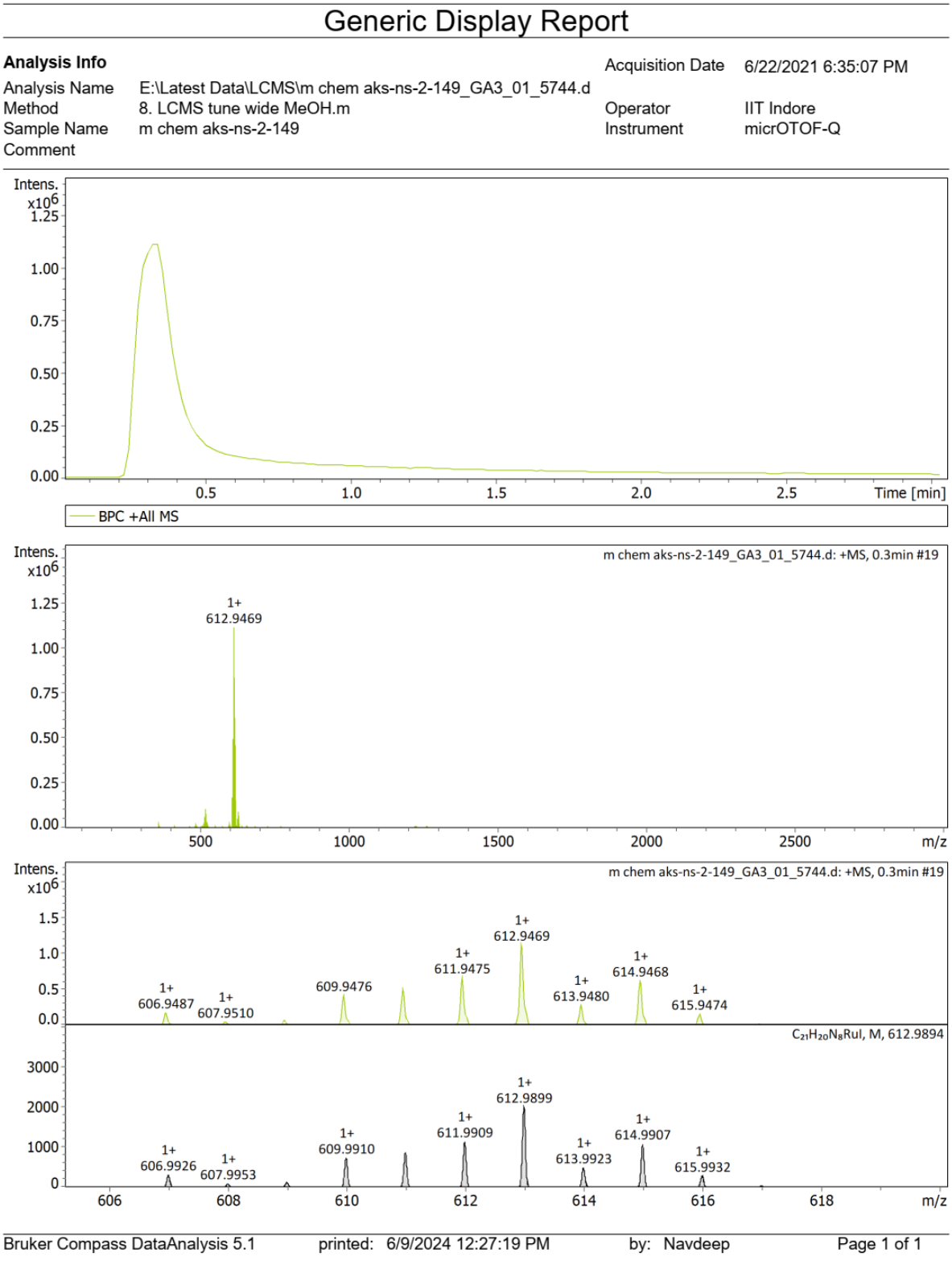


Figure S7. ESI-MS of complex **3.**

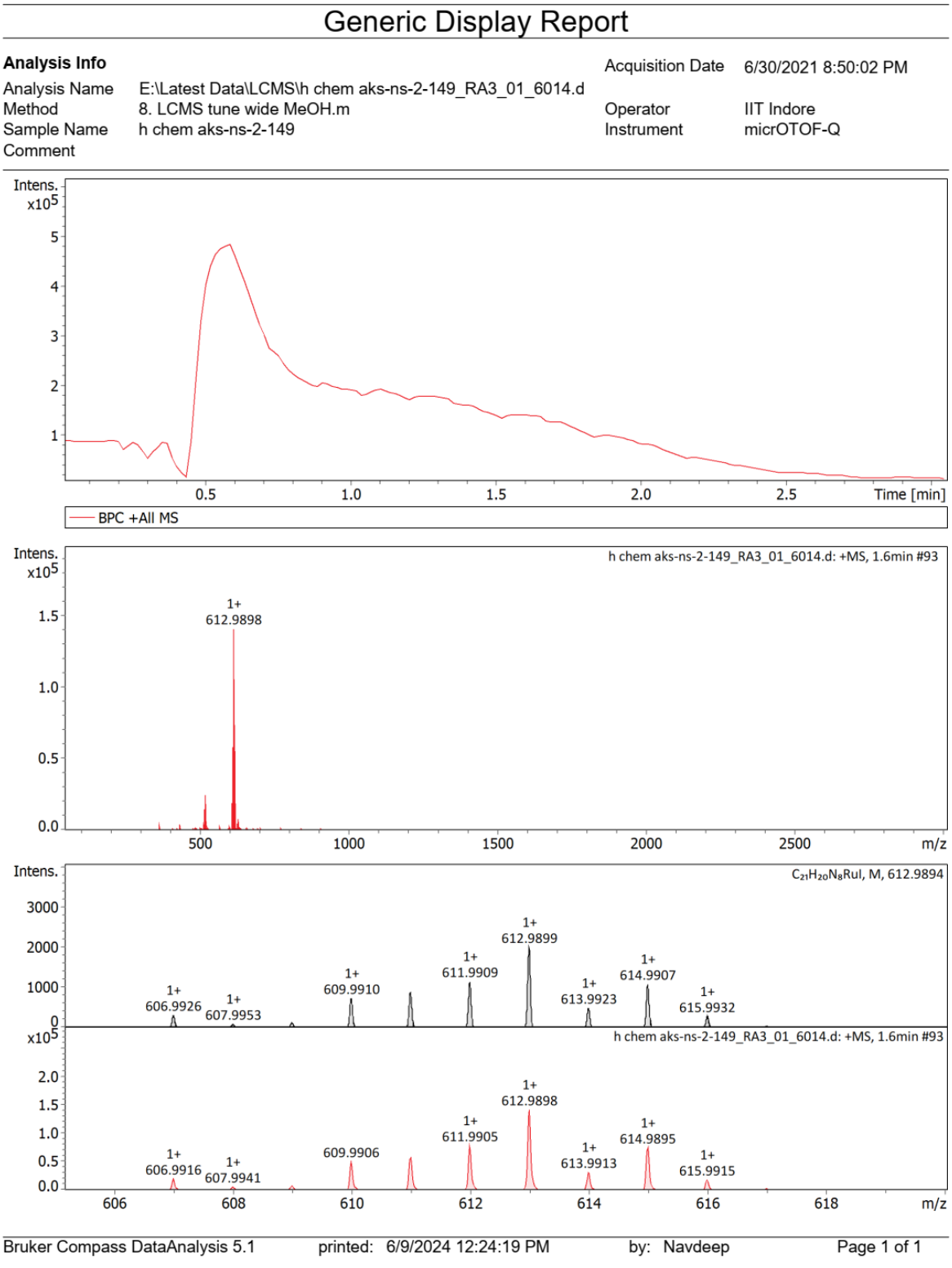


Figure S8. HRMS of complex **3.**

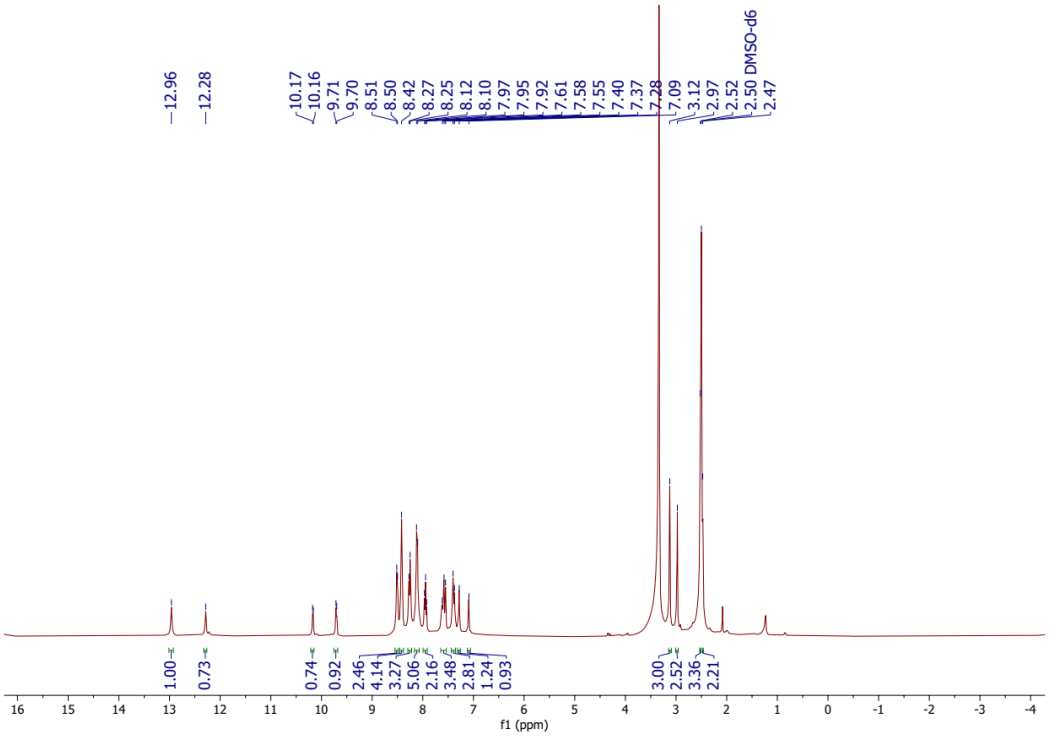


Figure S9. ^{1}H NMR of complex **3**

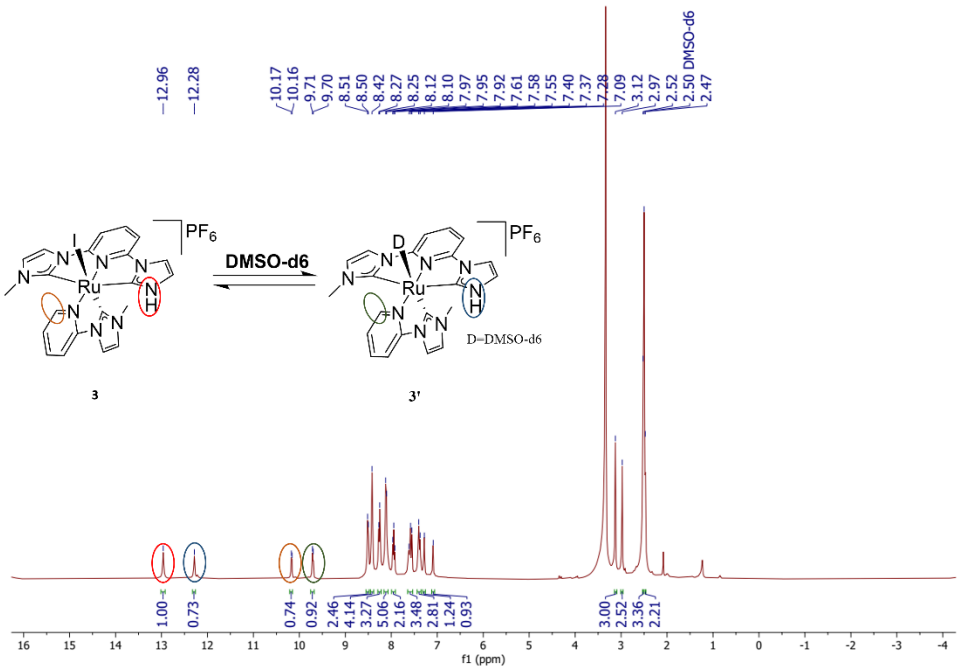


Figure S10. Equilibrium between 3 and 3' in the NMR tube in dmso-d6.

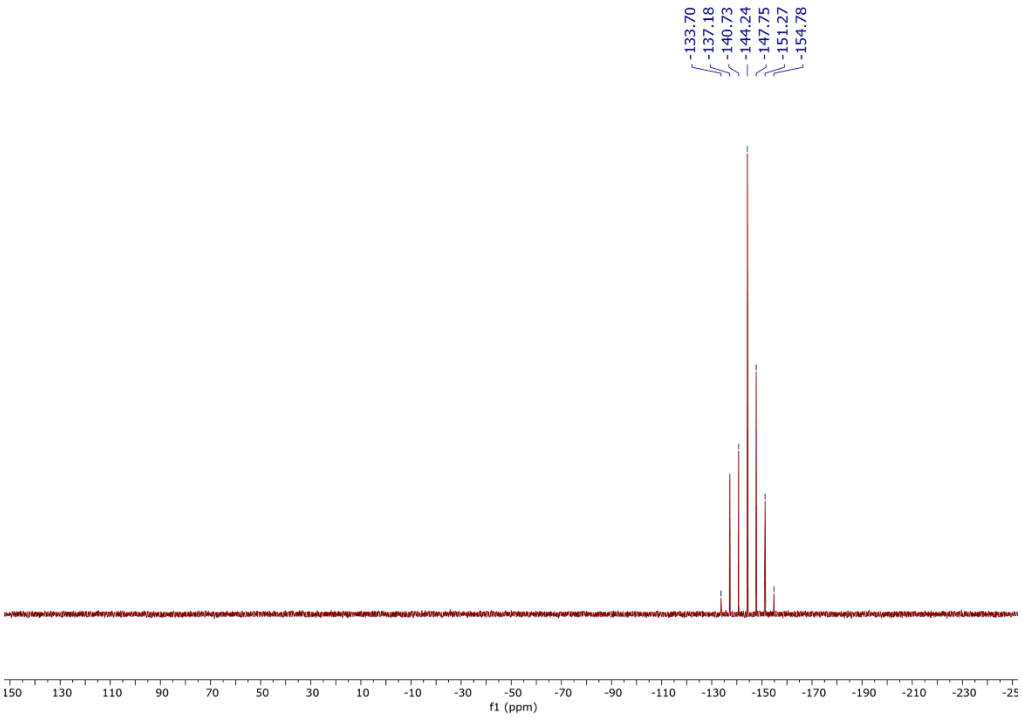


Figure S11. $^{31}P\{^{1}H\}$ NMR of complex **3**

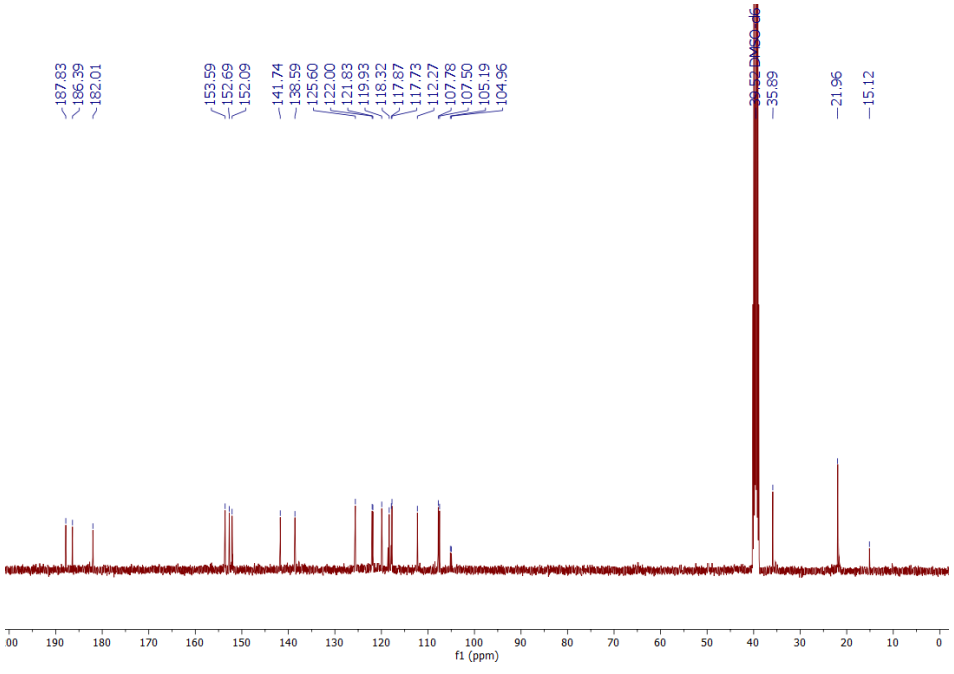


Figure S12. $^{13}C\{^{1}H\}$ NMR of complex **3**

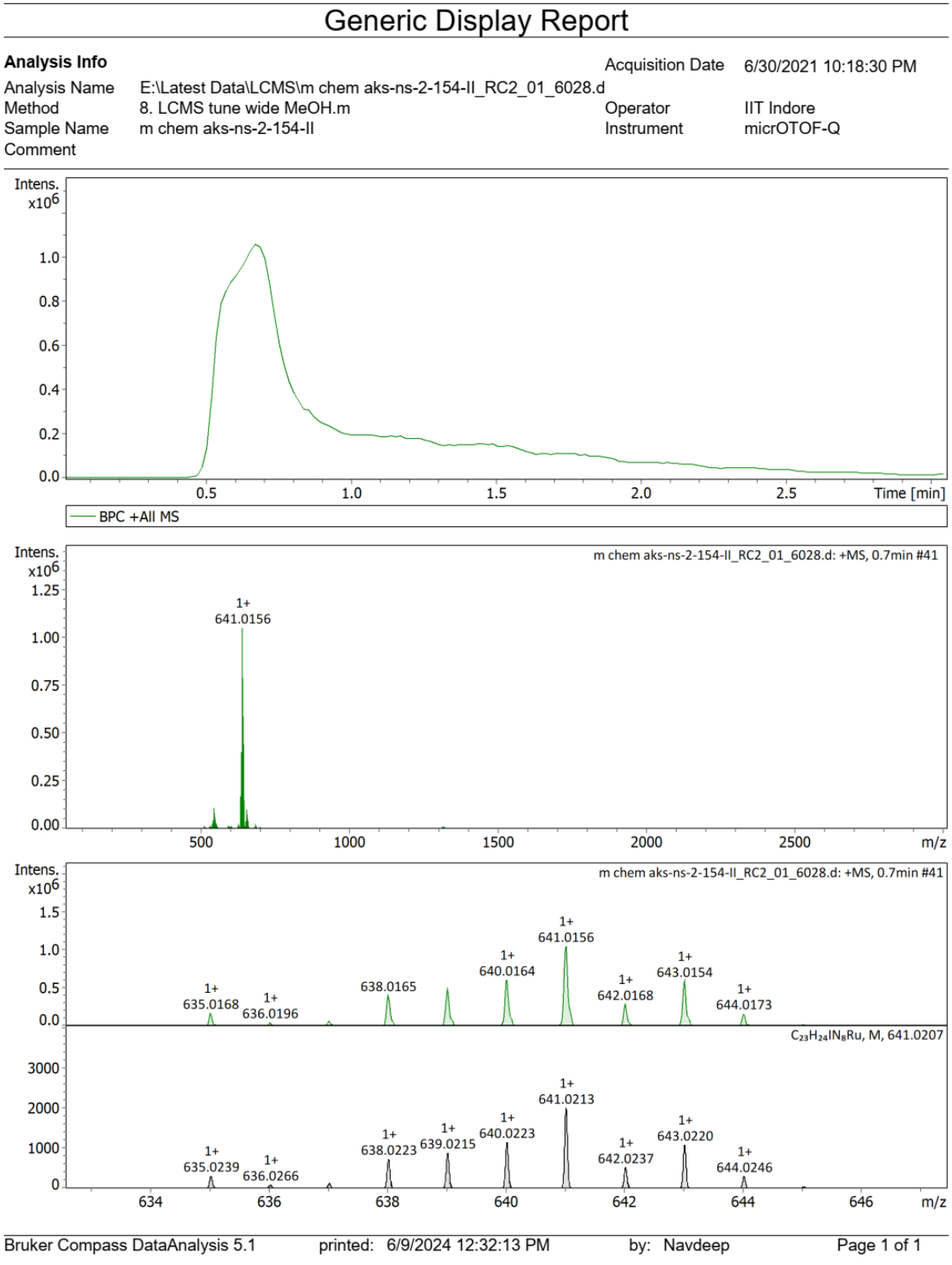


Figure S13. ESI-MS of complex **4**

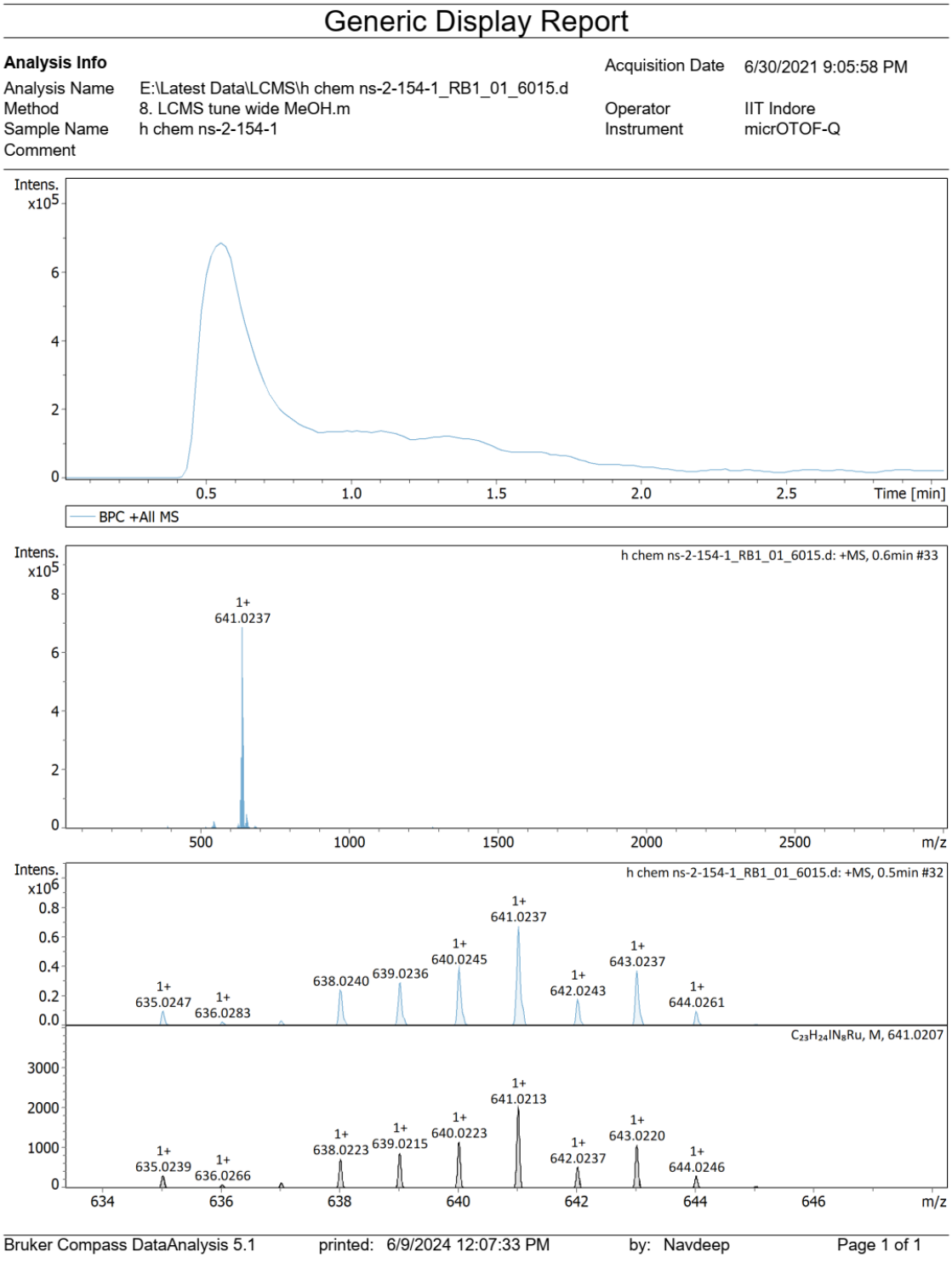


Figure S14. HRMS of complex **4**

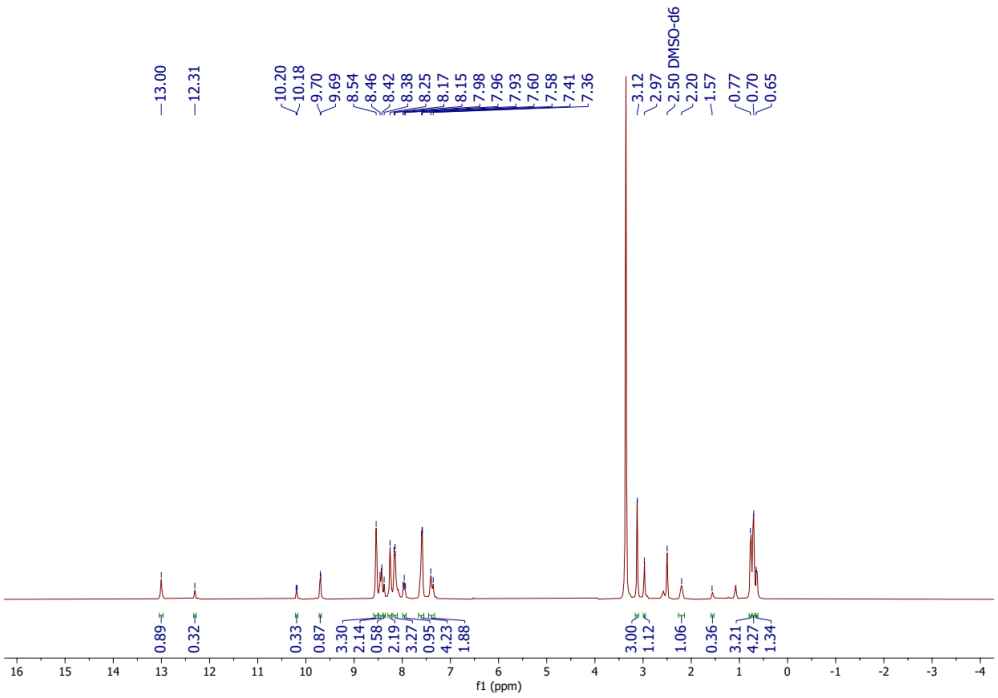


Figure S15. ^{1}H NMR of complex **4**

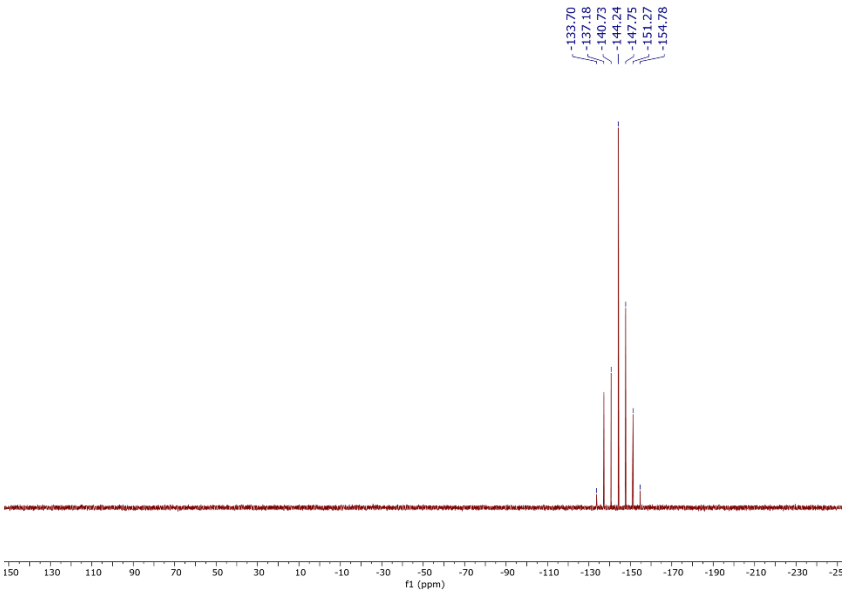


Figure S16. $^{31}P\{^{1}H\}$ NMR of complex **4**

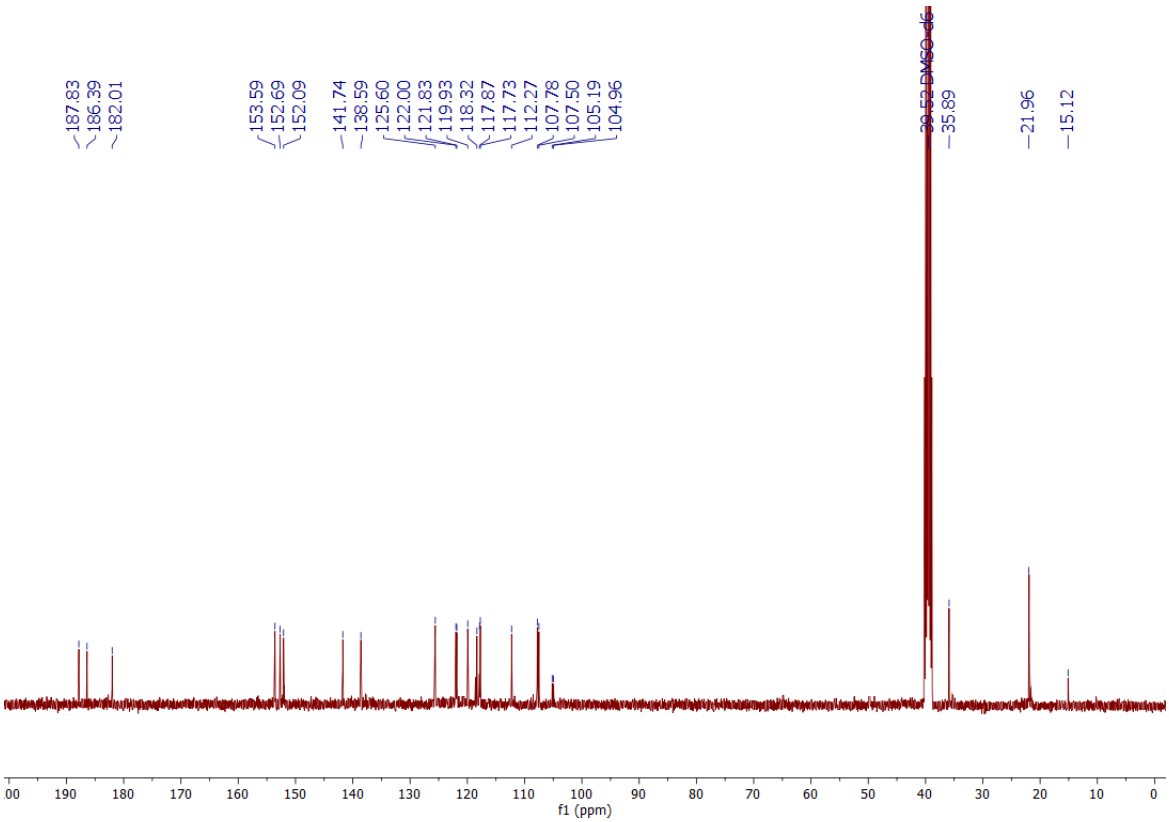


Figure S17. $^{31}C\{H\}$ NMR of complex **4**

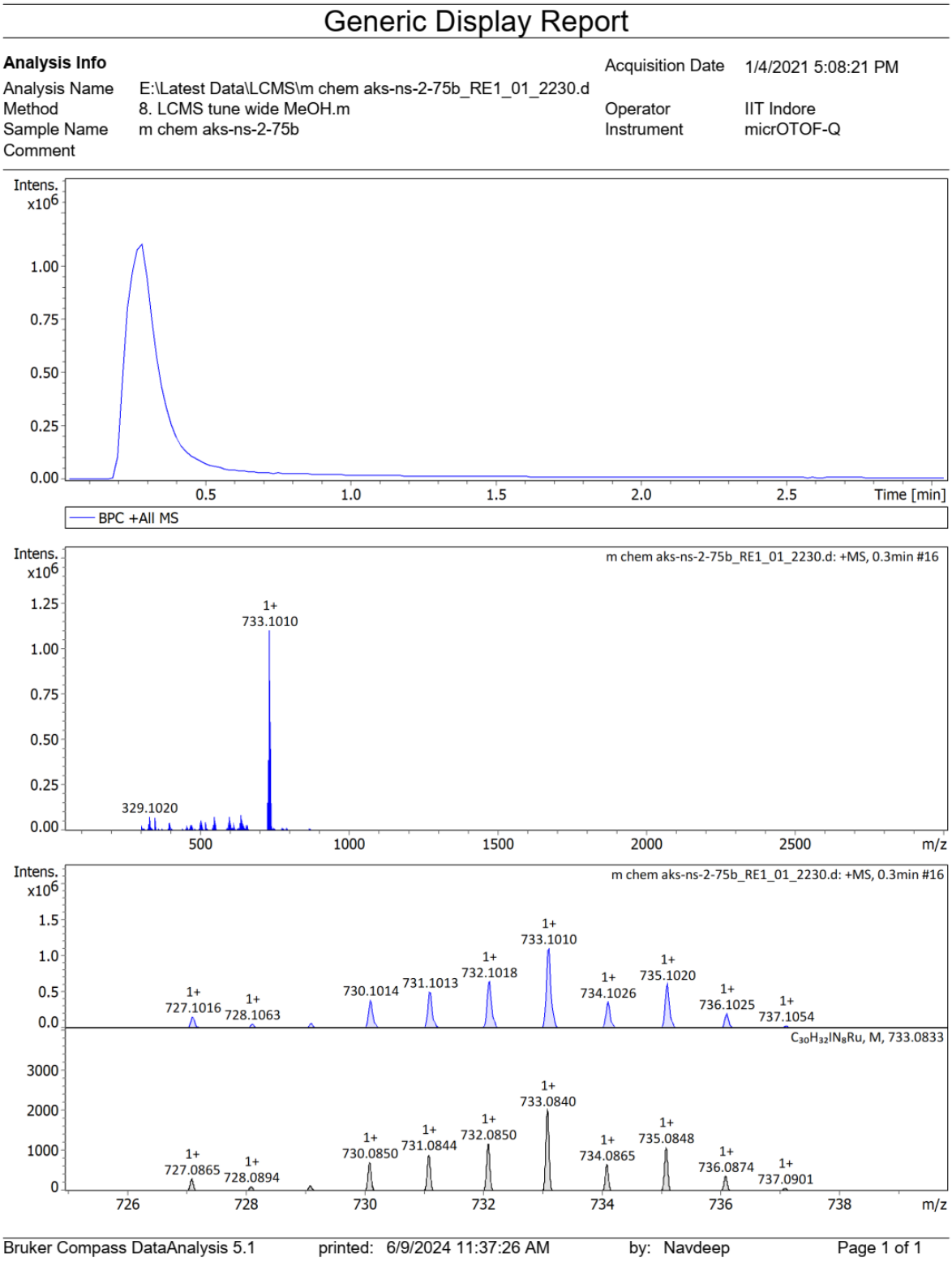


Figure S18. ESI-MS of complex **5**

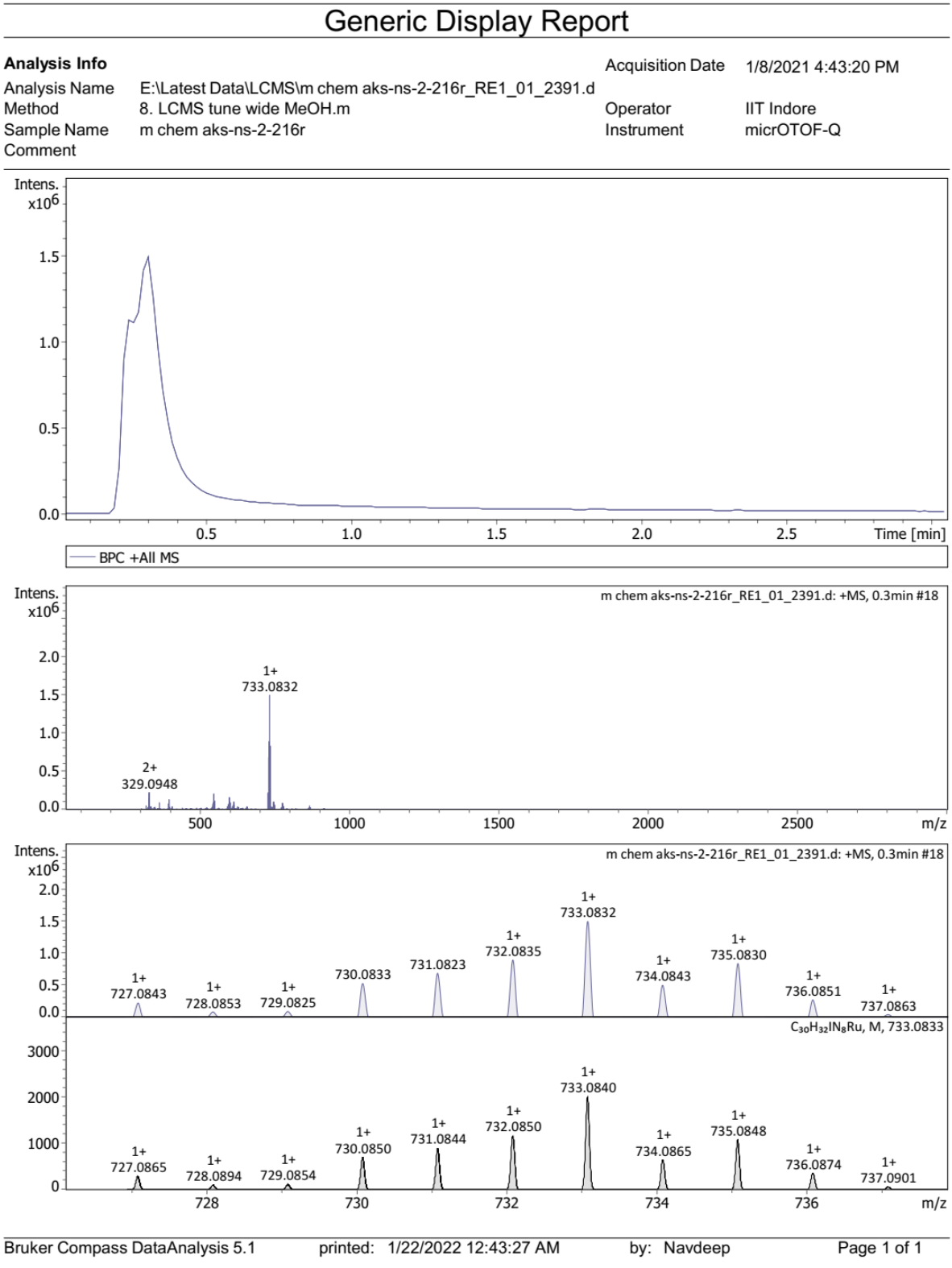


Figure S19. HRMS of complex **5**

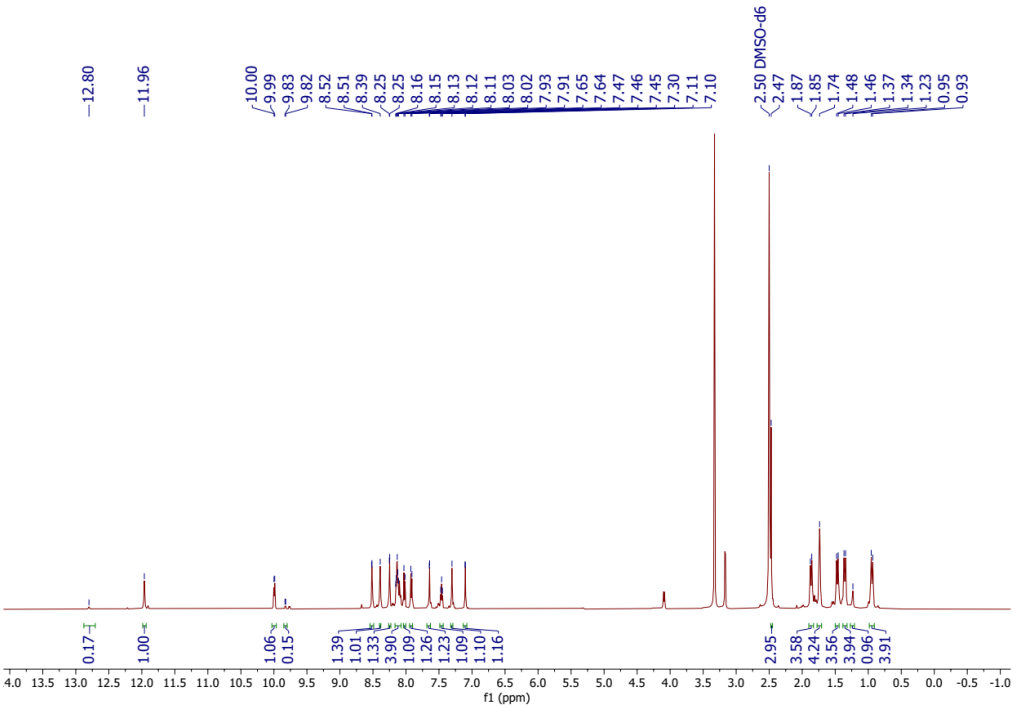


Figure S20. ^{1}H NMR of complex **5**

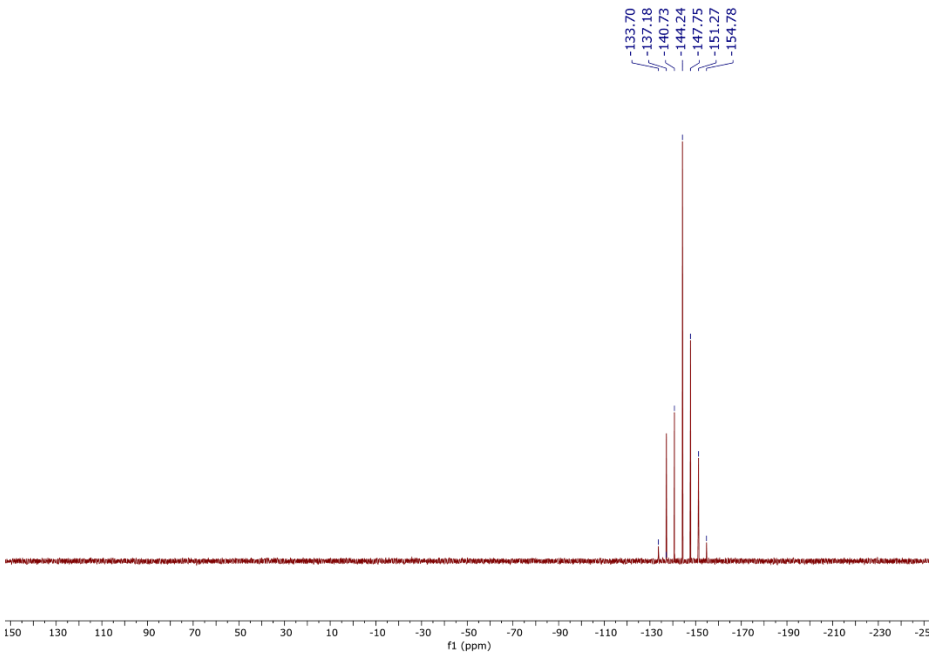


Figure S21. ^{31}P{^{1}H} NMR of complex **5**

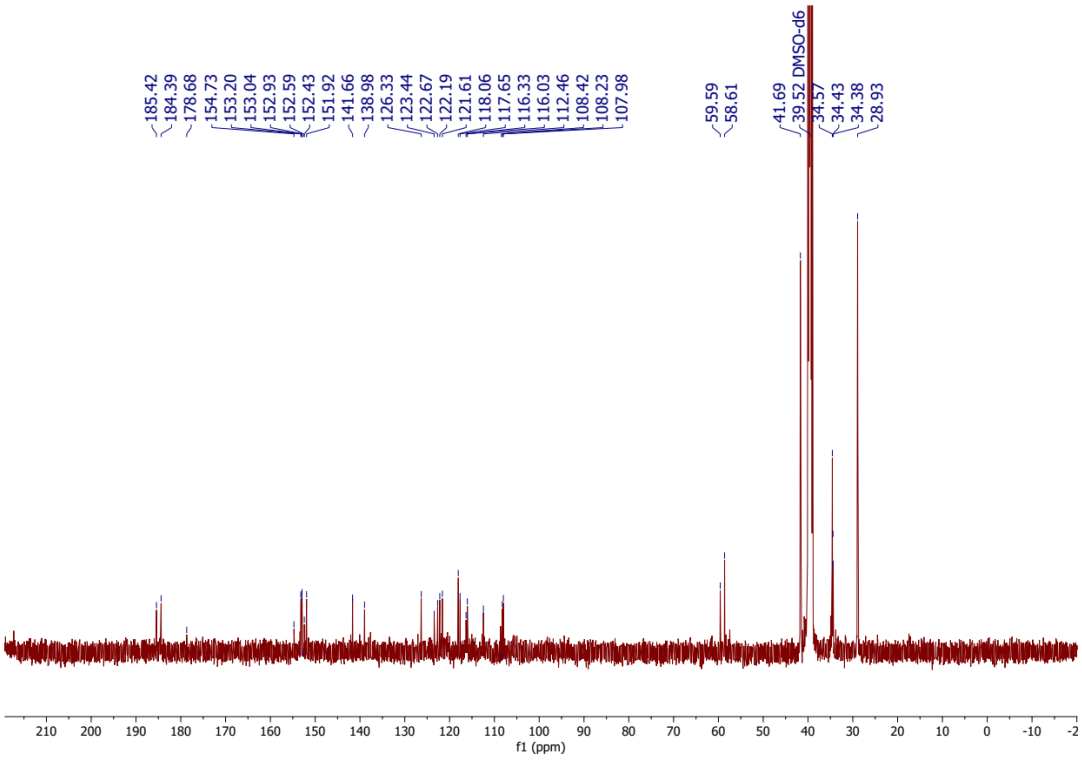


Figure S22. $^{13}C\{^{1}H\}$ NMR of complex **5**

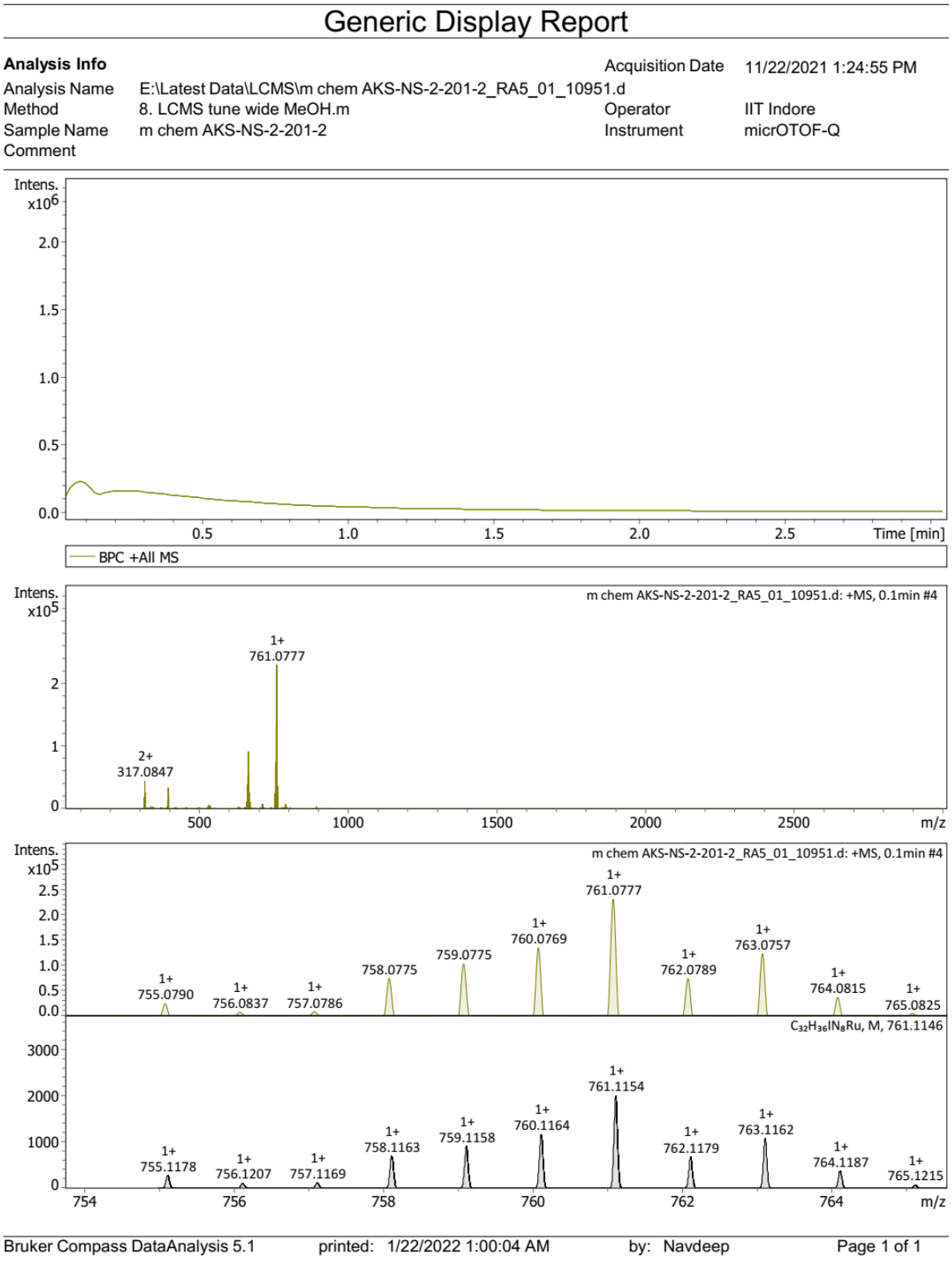


Figure S23. ESI-MS of complex **6**

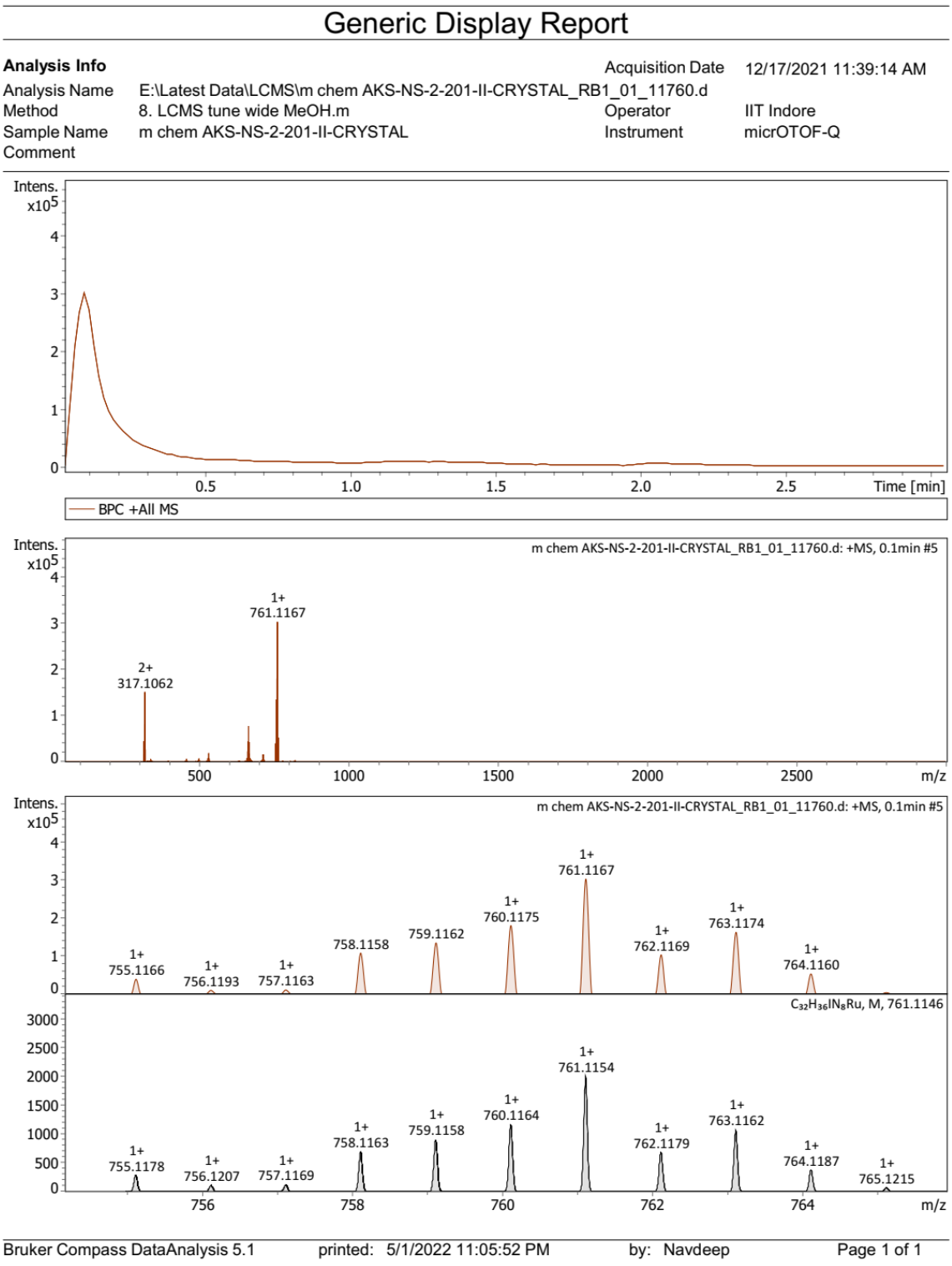


Figure S24. HRMS of complex **6**

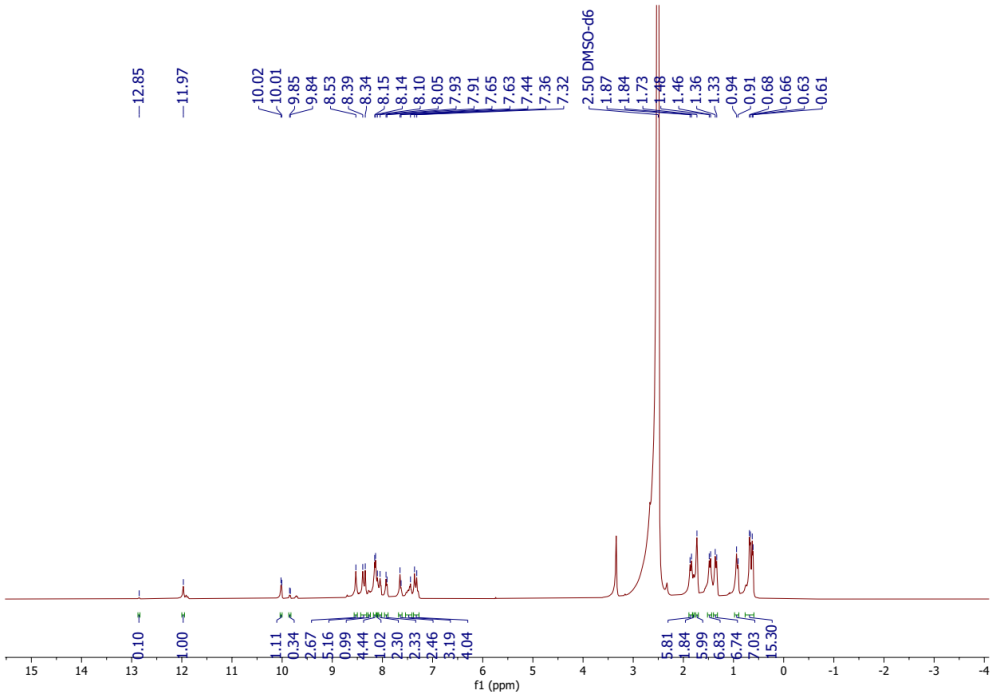


Figure S25. ^{1}H NMR of complex **6**

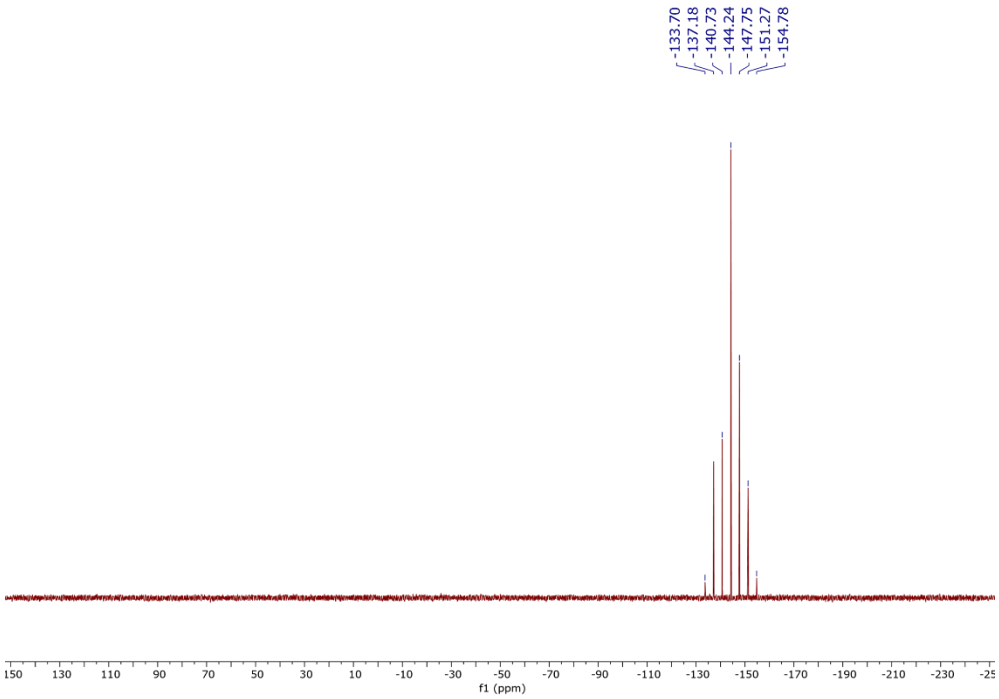


Figure S26. ^{31}P{^{1}H} NMR of complex **6**

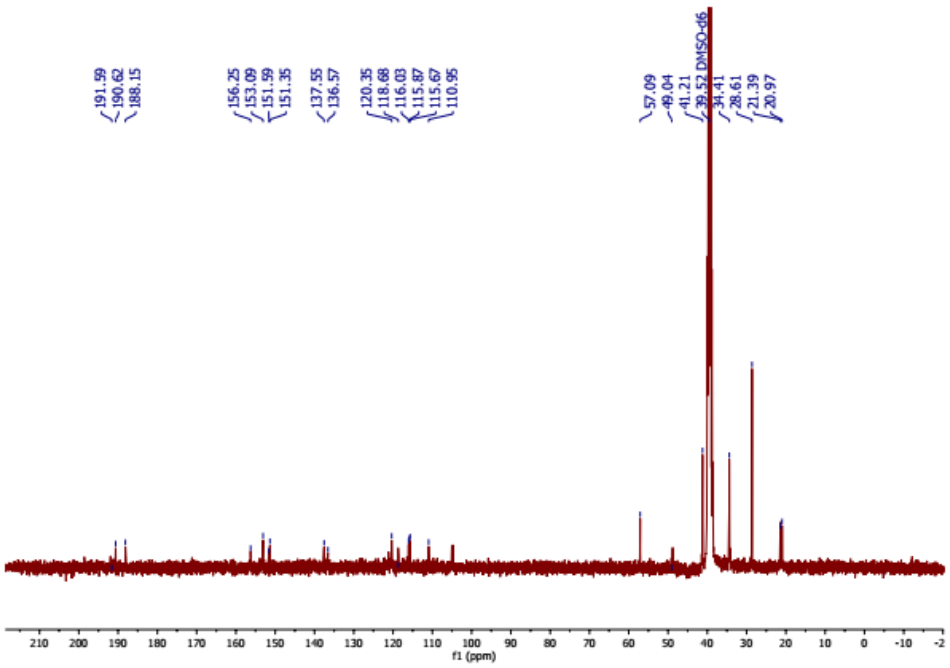


Figure S27. $^{13}C\{^{1}H\}$ NMR of complex **6**

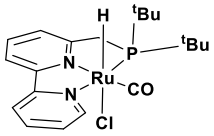
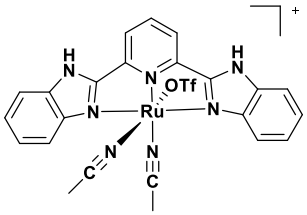
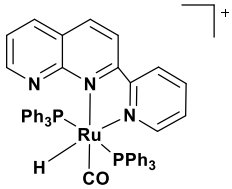
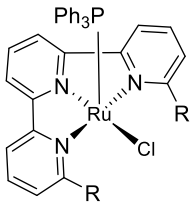
Table S1. Crystal data and structure refinement parameters of complex **6**

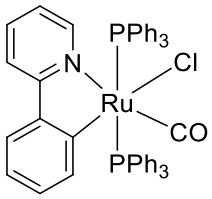
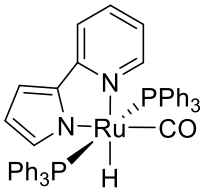
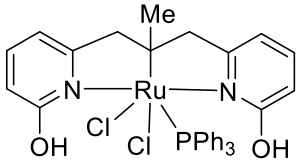
Empirical formula	**$C_{33}H_{40}Cl_{0.14}F_6I_{0.85}N_8OPRu$**
Formula weight	**924.41**
Temperature/K	**293(2)**
Crystal system	**triclinic**
Space group	**P-1**
a/Å	**11.7722(3)**
b/Å	**12.5423(3)**
c/Å	**14.3998(4)**
α/°	**77.402(2)**
β/°	**69.513(2)**
γ/°	**71.130(2)**
Volume/Å^3	**1871.37(9)**
Z	**2**
ρ_{calc}g/cm^3	**1.641**
μ/mm^{-1}	**1.246**
F(000)	**926.0**
Crystal size/mm^3	**0.25 × 0.2 × 0.18**
Radiation	**MoKα (λ = 0.71073)**
2Θ range for data collection/°	**6.632 to 58.01**
Index ranges	**$-15 \le h \le 15$, $-16 \le k \le 16$, $-18 \le l \le 14$**
Reflections collected	**20121**
Independent reflections	**8714 [R_{int} = 0.0484, R_{sigma} = 0.0653]**
Data/restraints/parameters	**8714/1/474**
Goodness-of-fit on F^2	**1.044**
Final R indexes [I>=2σ (I)]	**R_1 = 0.0477, wR_2 = 0.1048**
Final R indexes [all data]	**R_1 = 0.0712, wR_2 = 0.1189**
Largest diff. peak/hole / e Å^{-3}	**0.77/-0.67**

Table S2. Selected bond lengths and bond angles of complex **6**

Complex	Bond lengths (Å)		Bond Angles (°)	
6				
	Ru1-I1	2.7971(7)	N3-Ru1-I1	87.32(9)
	Ru1-N3	2.007(3)	N3-Ru1-N6	170.60(11)
	Ru1-N6	2.121(3)	N3-Ru1-C1	78.01(13)
	Ru1-C1	2.031(3)	N3-Ru1-C9	77.80(12)
	Ru1-C9	2.119(3)	N3-Ru1-C29	100.47(15)
	Ru1-C29	1.972(4)	N6-Ru1-I1	94.36(9)
	N1-C1	1.342(4)	N6-Ru1-C1	92.74(12)
	N2-C1	1.396(4)	N6-Ru1-C9	111.47(12)
	N5-C9	1.348(4)	N6-Ru1-C29	78.27(15)
	N4-C9	1.392(4)	N1-C1-N2	103.0(3)
	N8-C29	1.338(5)	N5-C9-N4	103.4(3)
	N7-C29	1.392(5)	N8-C29-N7	103.2(4)
	Ru1-Cl1	2.740(16)	N3-Ru1-Cl1	81.6(6)
	-	-	N8-Ru1-Cl1	99.6(6)

Table S3. Comparative study for catalytic dehydrogenation of benzyl alcohol to benzoic acid.

. Entry	Cat- (mol %)	temp. (°C)	Time (h)	Base (equiv.)	Solvent	TON/ (TOF)
1.	This work 4 (**0.01**) 4 (**0.005**)	 110 110	 24 120	 KOH (1) KOH (1)	 toluene toluene	 10,000 (416 h^{-1}) 20,000 (167 h^{-1})
2.	Milstein et al. (**0.2**) 	110	18	NaOH (1.1)	water	455/ ($25h^{-1}$)
3.	Peng et al. (**0.2**) 	150	24	CsOH (1.0)	-	400/ ($17h^{-1}$)
4.	Bera etal. (**5.0**) 	110	6	NaOH (18.5)	water	20/ ($3h^{-1}$)
5.	Szymczak et al. (**0.4**) 	120	18	KOH (3)	toluene	250/ ($14h^{-1}$)

6.	Chen et al (**0.5**)  	110	6	KOH (1.5)	toluene	200/ ($33h^{-1}$)
7.	Yi et al. (**0.1**)  	120	12	KOH (1.1)	toluene	4000/ ($333h^{-1}$)
8.	Daw (**1**)  	150	24	KOH (0.5)	Diglyme: H_2O (9:1)	400/ ($17h^{-1}$)

TON = [(Number of moles of substrate converted)/(Number of moles of catalyst)] at the end of the reaction.

TOF = [(TON)/hour]

Mechanistic Investigation.

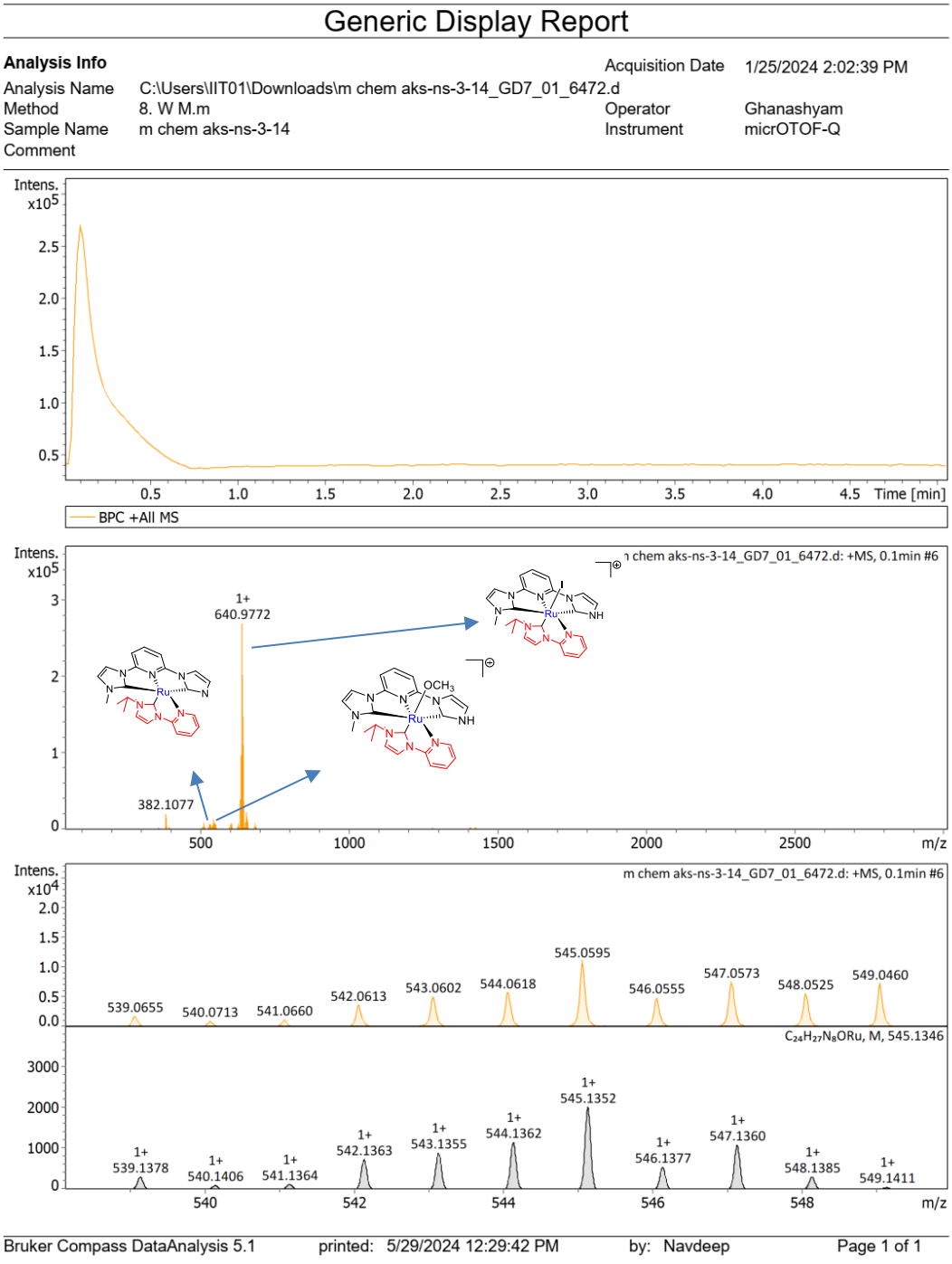


Figure S28. LCMS of catalyst 4 with KOH (2 equiv.) after 1 h.

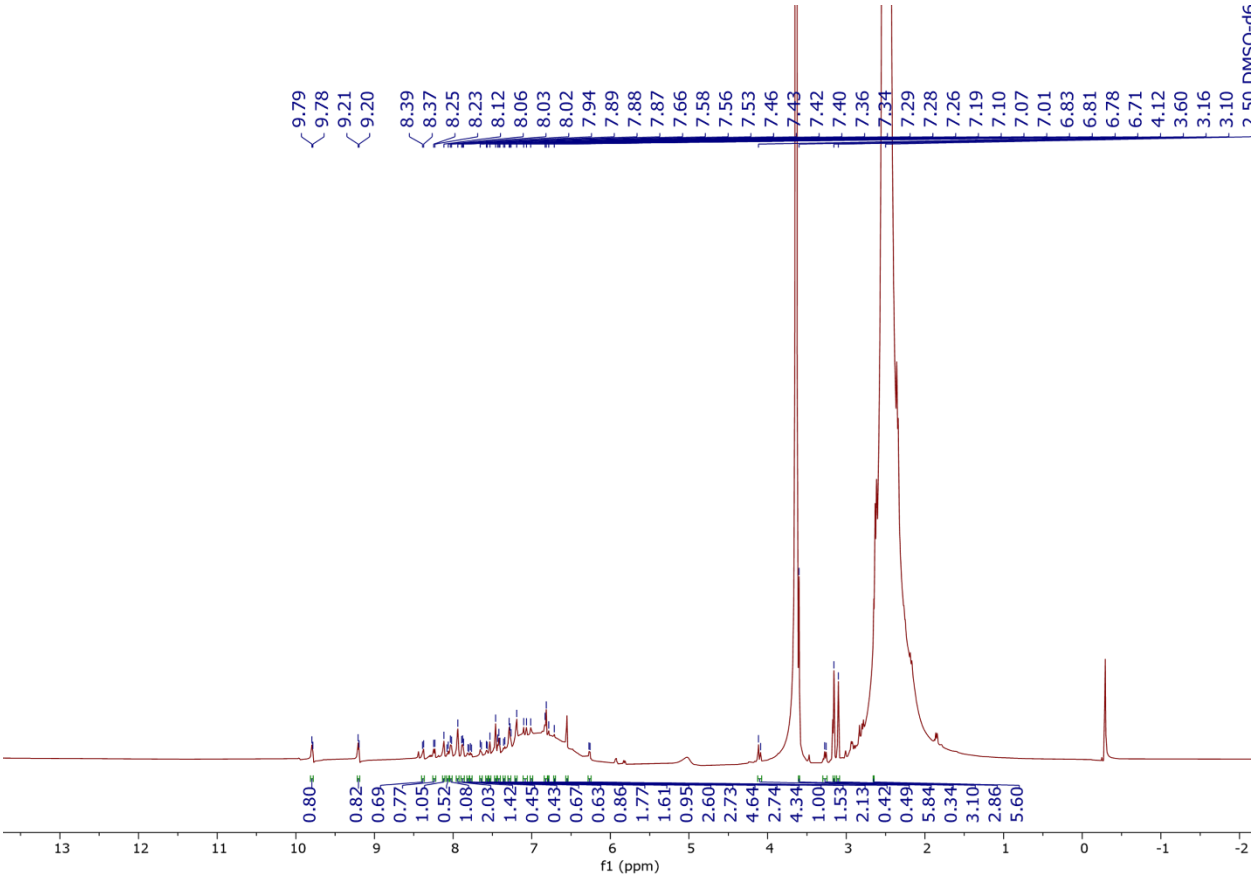


Figure S29. Deprotonation of complex **4** with KOH in NMR tube

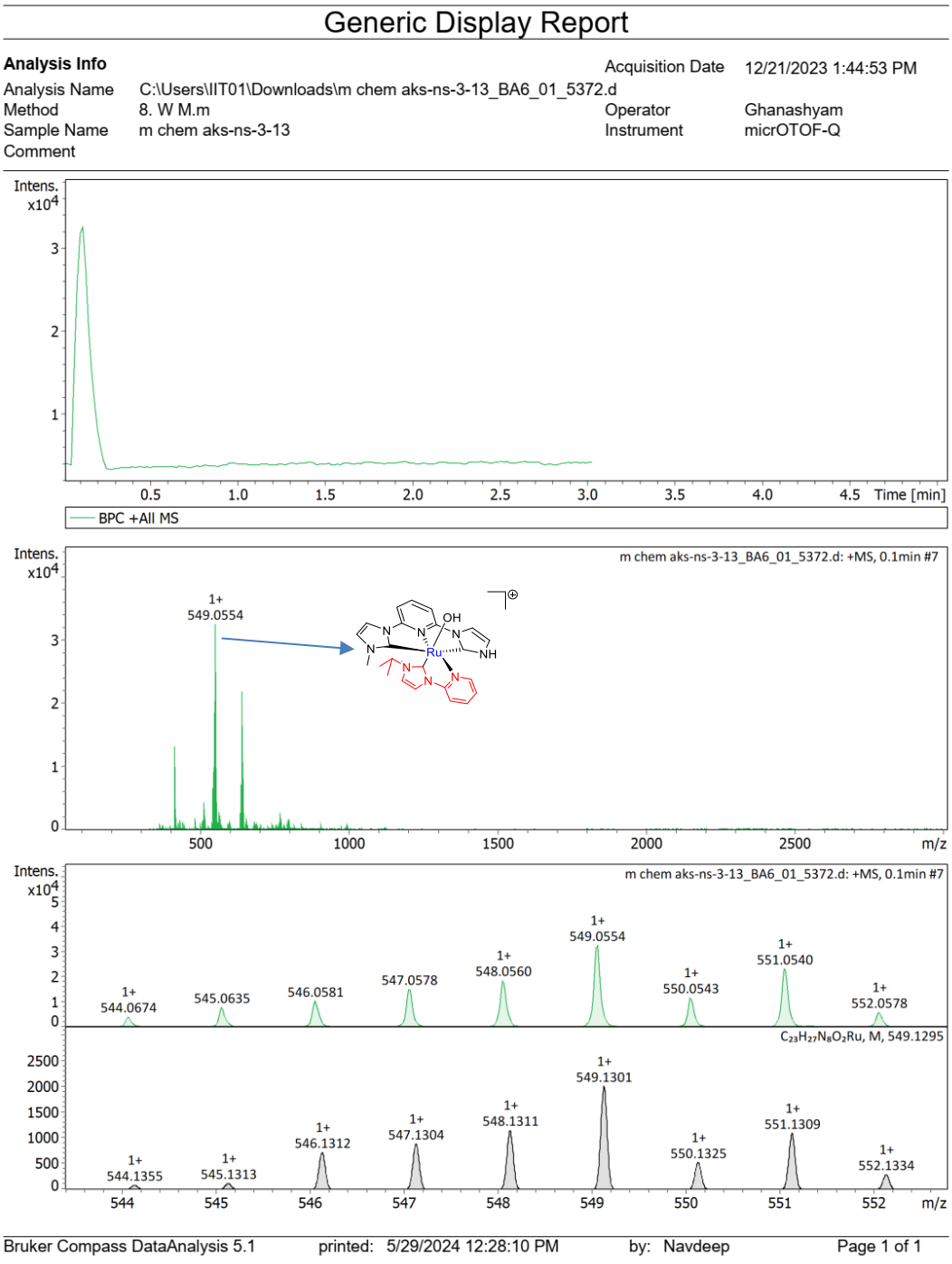


Figure S30. LCMS of catalyst 4 with KOH (2 equiv.) in toluene after 2 h.

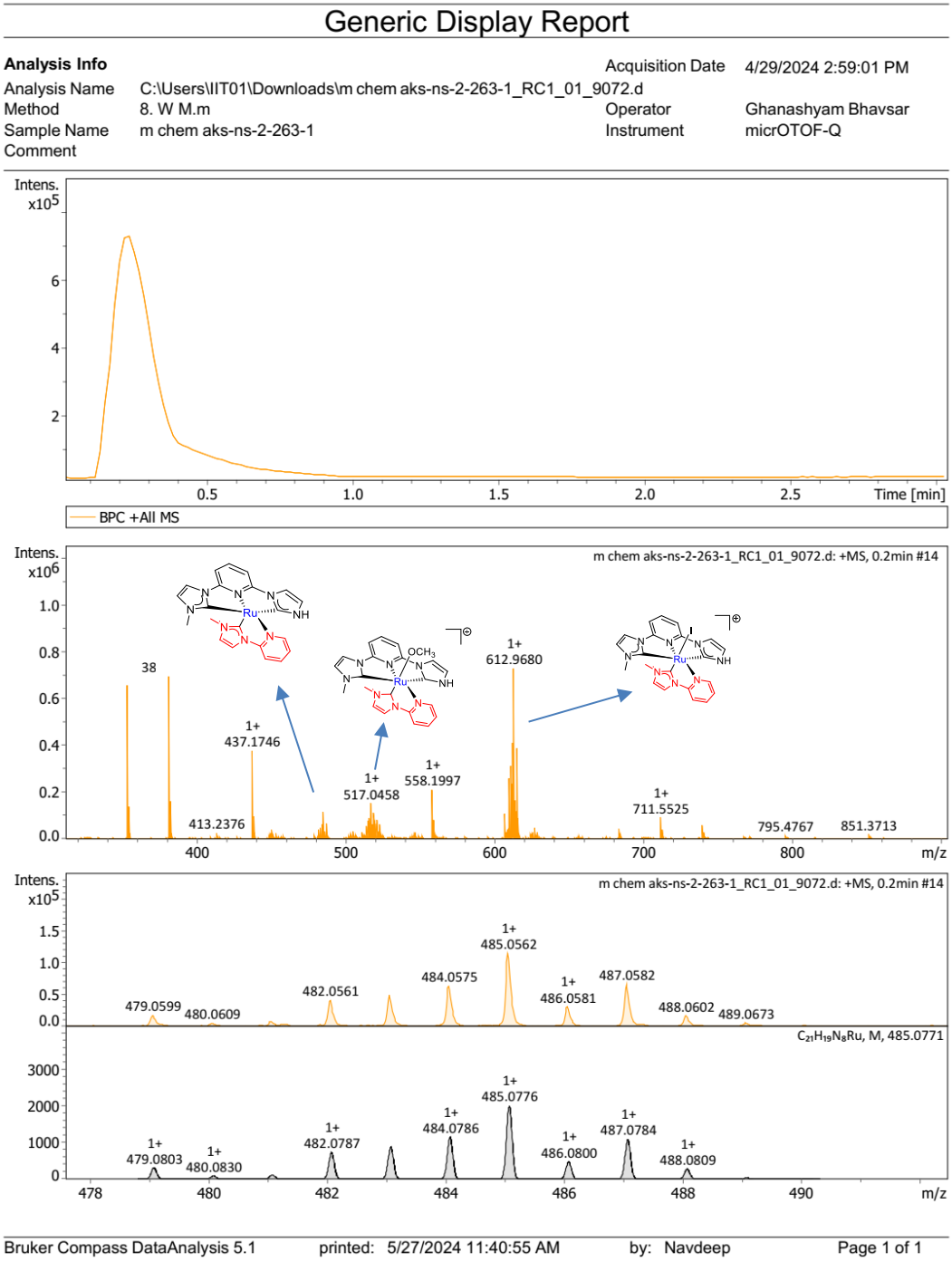


Figure S31 . LCMS of reaction mixture of cat 3 and KOH in toluene (2 equiv.)

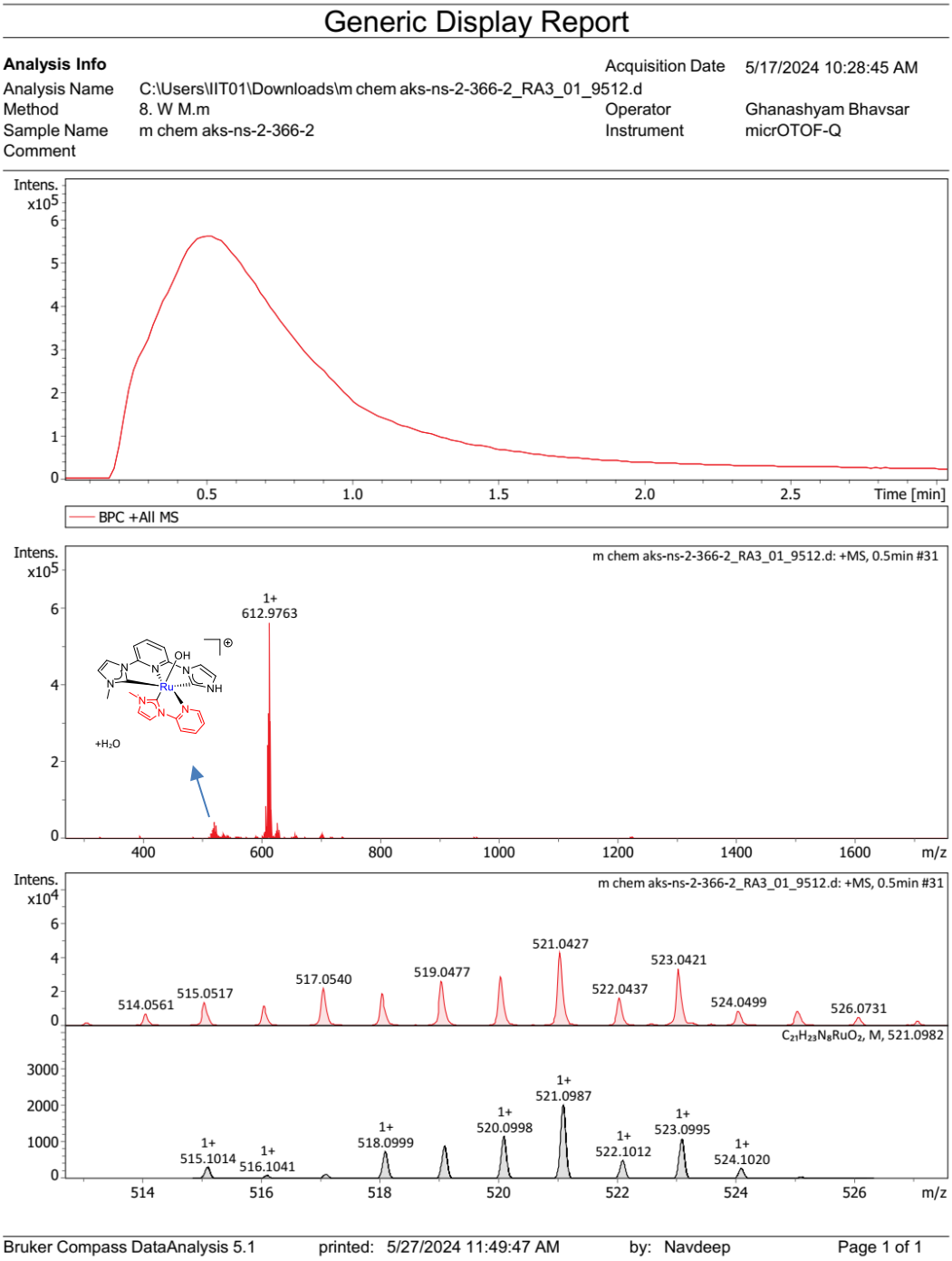


Figure S32 . LCMS of reaction mixture of cat 3 and KOH in toluene (1 equiv.)

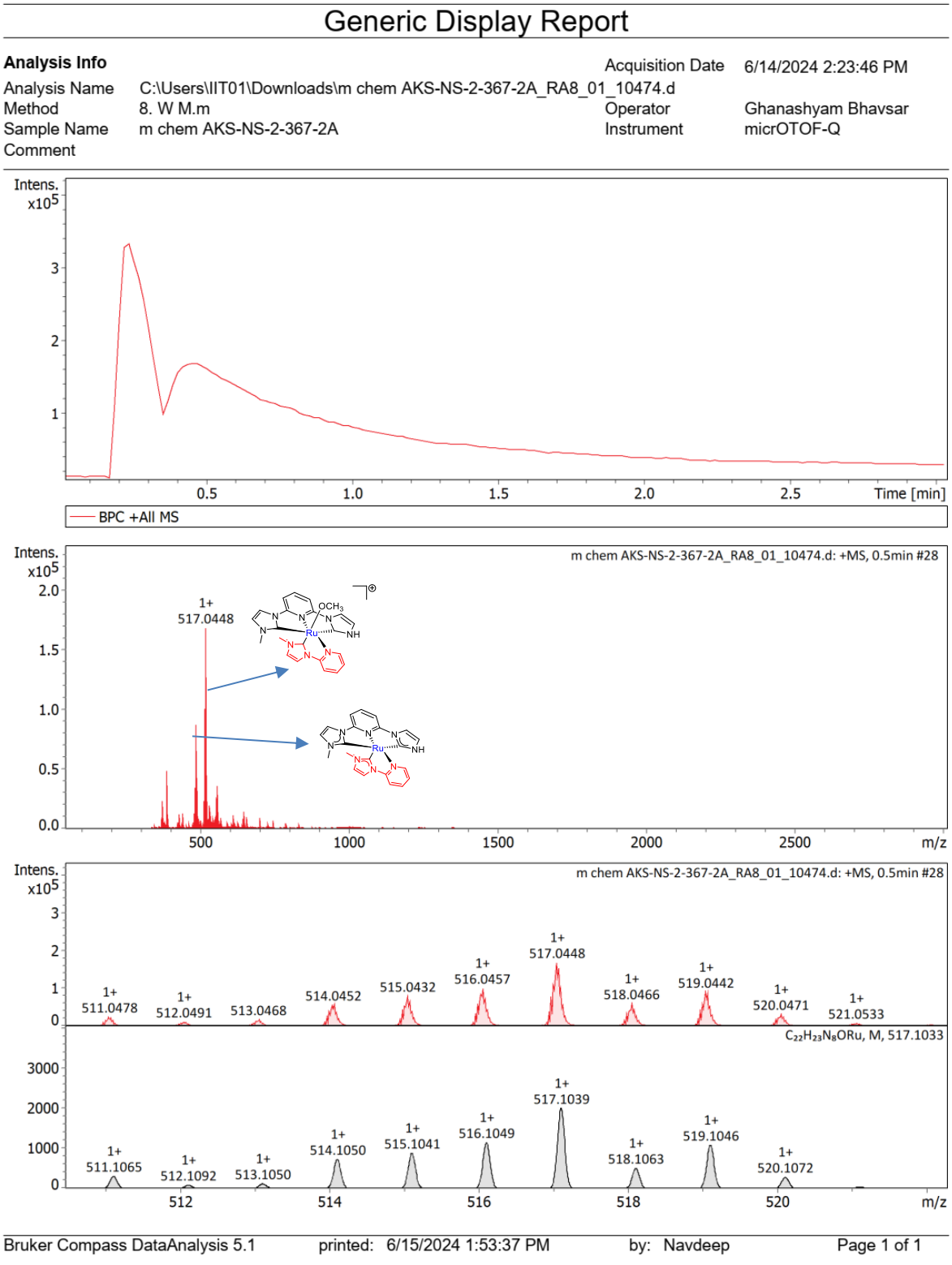


Figure S33 . LCMS of reaction mixture of cat 3 and KOH in water.

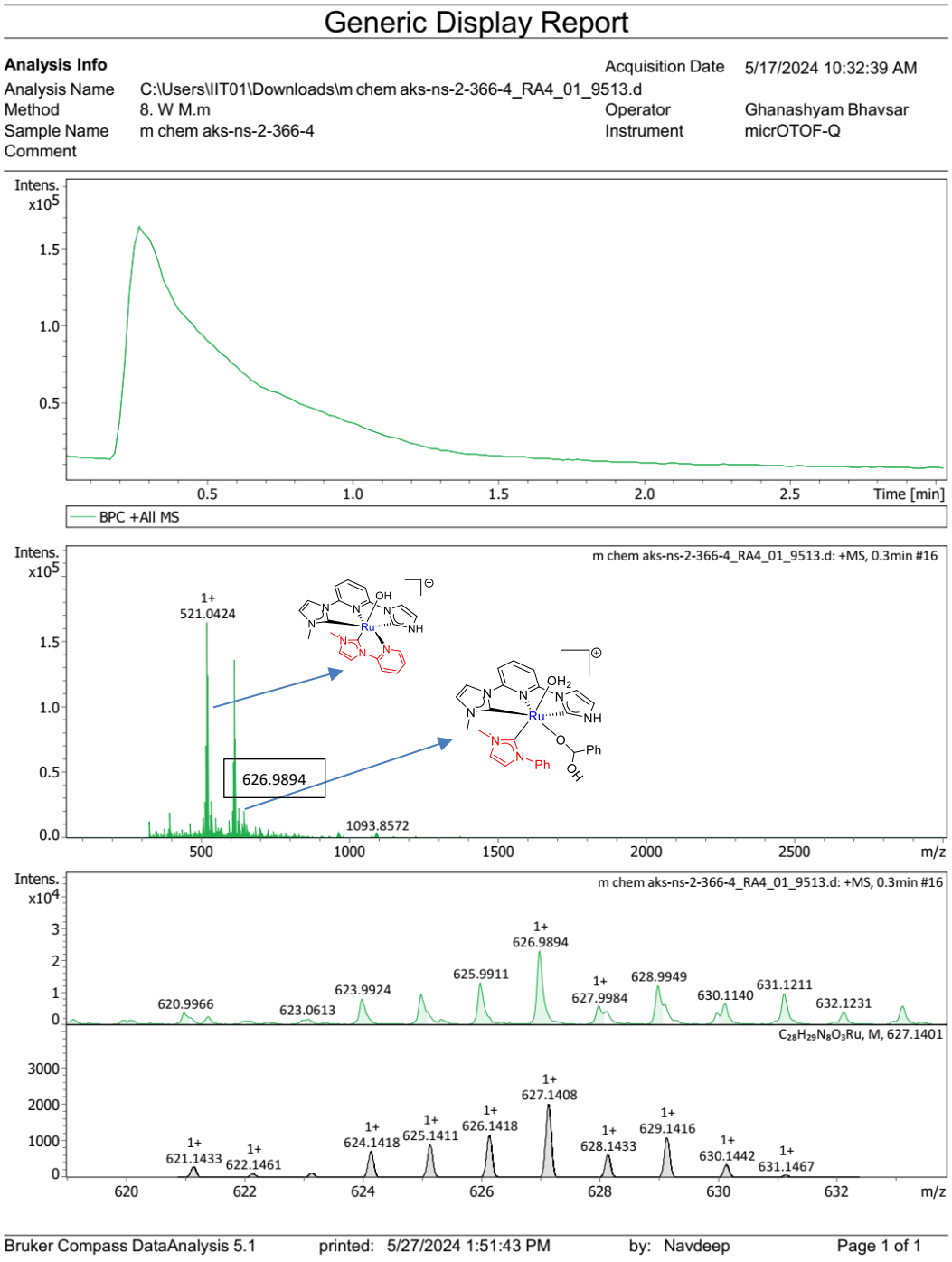


Figure S34 . LCMS of reaction mixture of after addition of benzyl alcohol substrate.

Dehydrogenation of benzaldehyde to benzoic acid

Catalyst **4** (0.1 mol%), KOH (1 mmol) were mixed in dry toluene 3 ml in a schlenk tube and then stirred for 1 hour. After that the oil bath temperature was heated to 120 °C and benzaldehyde (1 mmol) was added to reaction mixture and schlenk tube was dipped in preheated oil bath under inert atmosphere. The reaction was stirred for 1 hour and cooled. The workup was followed as mentioned in procedure. The product was analysed by ^{1}H-NMR showing formation of benzoic acid with traces of benzyl alcohol.

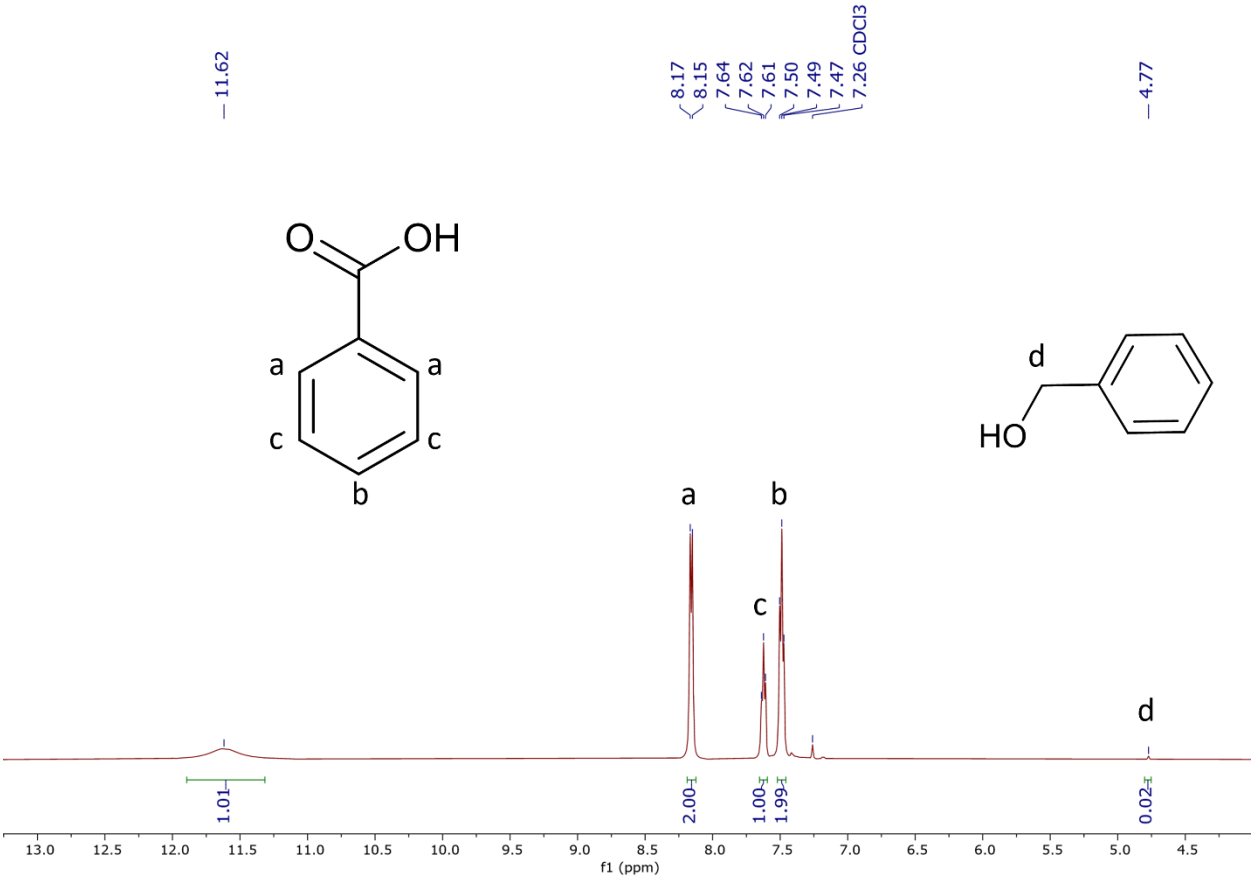


Figure S35. NMR spectra for dehydrogenation of benzaldehyde

NMR of carboxylic acid products after dehydrogenation of alcohol

(4a) Benzoic acid:

^{1}H NMR (500 MHz, $CDCl_3$) δ 11.67 (s, 1H), 8.14 (dd, *J* = 1.5, 8.4 Hz, 2H), 7.63 (t, *J* = 7.4 Hz, 1H), 7.49 (t, *J* = 7.8 Hz, 2H). ^{13}C{^{1}H} NMR (126 MHz, CDCl3) δ 172.18 (COOH), 133.72, 130.12, 129.20, 128.39.

(4b) p-Methoxy benzoic acid:

^{1}H NMR (500 MHz, $CDCl_3$) δ 6.39 (dd, *J* = 5.5, 8.9 Hz, 2H), 6.13 (t, *J* = 8.9 Hz, 2H), 2.94 (s, 3H). ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 167.18 (COOH), 163.01, 131.52, 123.14, 113.99, 55.62.

(4c) m-Methoxy benzoic acid

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.72 (d, *J* = 7.6 Hz, 1H), 7.63 (s, 1H), 7.39 (t, *J* = 8.0 Hz, 1H), 7.17 (ddd, *J* = 1.0, 2.7, 8.3 Hz, 1H), 3.87 (s, 3H). ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 171.69 (COOH), 159.77, 130.65, 129.69, 122.83, 120.63, 114.54, 55.62.

(4d) o-Methoxy benzoic acid

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.06 (dd, *J* = 1.5, 8.3 Hz, 1H), 7.45 (td, *J* = 1.5, 7.5 Hz, 1H), 7.27 (m, 2H), 2.58 (s, 3H). ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 172.55 (COOH), 141.50, 133.09, 132.08, 131.71, 128.34, 126.00, 29.85.

(4e) m,p-dimethoxybenzoic acid

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.78 (dd, *J* = 2.0, 8.4 Hz, 1H), 7.60 (d, *J* = 2.1 Hz, 1H), 6.92 (d, *J* = 8.5 Hz, 1H), 3.95 (d, *J* = 5.2 Hz, 6H). ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 171.71 (COOH), 153.86, 148.83, 124.73, 121.81, 112.45, 110.46, 56.16.

(4f) p-(benzyloxy)benzoic acid

^{1}H NMR (500 MHz, DMSO) δ 12.62 (s, 1H), 7.92 – 7.86 (m, 2H), 7.46 (d, *J* = 7.1 Hz, 2H), 7.37 (dt, *J* = 7.4, 29.5 Hz, 3H), 7.09 (d, *J* = 8.1 Hz, 2H), 5.18 (d, *J* = 3.2 Hz, 2H). ^{13}C{^{1}H} NMR (126 MHz, DMSO) δ 166.97 (COOH), 161.93, 136.54, 131.34, 128.50, 127.82, 114.62, 69.45.

(4g) p-Methyl benzoic acid:

^{1}H NMR (500 MHz, DMSO) δ 7.83 (d, *J* = 8.1 Hz, 2H), 7.30 (d, *J* = 8.2 Hz, 2H), 2.37 (s, 3H). ^{13}C{1H} NMR (126 MHz, DMSO) δ 167.16 (COOH), 142.82, 129.16, 127.94, 20.96.

(4h) m-Methyl benzoic acid:

^{1}H NMR (500 MHz, DMSO) δ 12.83 (s, 1H), 7.78 – 7.71 (m, 2H), 7.41 (dd, *J* = 7.7, 22.6 Hz, 2H), 2.36 (s, 3H). ^{13}C{^{1}H} NMR (126 MHz, DMSO) δ 167.43, 137.91, 133.48, 130.73, 129.74, 128.47, 126.47, 20.83.

(4i) p-^{Iso}Propyl benzoic acid:

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.04 (d, *J* = 6.3 Hz, 2H), 7.33 (d, *J* = 8.4 Hz, 2H), 3.04 – 2.93 (m, 1H) 1.27 (d, *J* = 17.1 Hz, 6H). ^{13}C{1H} NMR (126 MHz, $CDCl_3$) δ 171.83, 155.40, 130.53, 127.16, 126.75, 34.49, 23.83.

(4j) p-^{tert}Butyl benzoic acid

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.7 Hz, 2H), 7.49 (d, *J* = 8.7 Hz, 2H), 1.36 (s, 9H). ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 171.95, 157.73, 130.27, 126.65, 125.64, 35.35, 31.25.

(4k) p-Amino benzoic acid

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.7 Hz, 2H), 6.66 (d, *J* = 8.7 Hz, 2H). ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 171.10 (COOH), 151.62, 132.53, 118.77, 113.93.

(4l) m-aminobenzoic acid

^{1}H NMR (500 MHz, DMSO) δ 12.55 (s, 1H) 7.16 (s, 1H), 7.13 – 7.04 (m, 2H), 6.79 – 6.73 (m, 1H). ^{13}C{^{1}H} NMR (126 MHz, DMSO) δ 167.90 (COOH), 148.85, 131.33, 128.89, 117.99, 116.64, 114.44.

(4m) o-aminobenzoic acid

No reaction

(4n) m- Nitro benzoic acid:

^{1}H NMR (500 MHz, DMSO-d_6): δ 8.29 (d, J = 8.4 Hz, 1H), 8.16 (t, J = 8.5 Hz, 1H), 7.9 (d, J = 8.5 Hz, 1H), 7.3 (s, J = 8.5 Hz, 1H). ^{13}C{1H}NMR (126 MHz, DMSO) δ 192.83, 130.93, 130.73, 127.09, 123.74, 123.53, 123.35.

(4o) p-Fluoro benzoic acid:

^{1}H NMR (500 MHz, DMSO) δ 8.00 (dd, J = 5.6, 8.9 Hz, 2H), 7.32 (t, J = 8.9 Hz, 2H). ^{13}C{1H} NMR (126 MHz, DMSO) δ 166.82, 131.87, 131.47, 130.30, 127.01.

(4p) p-Chloro benzoic acid:

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.03 (d, J = 4.4 Hz, 2H), 7.45 (d, J = 9.0 Hz, 2H). ^{13}C{^{1}H} NMR (126 MHz, DMSO) δ 166.82, 131.87, 131.47, 130.30, 127.01.

(4q) p-Bromo benzoic acid:

1H NMR (500 MHz, DMSO) δ 7.86 (d, J = 8.5 Hz, 2H), 7.70 (d, J = 6.4 Hz, 2H). ^{13}C{1H} NMR (126 MHz, DMSO) δ 166.82, 131.87, 131.47, 130.30, 127.01.

(4r) p-Iodo benzoic acid:

1H NMR (500 MHz, CDCl3) δ 8.03 (d, J = 4.4 Hz, 2H), 7.45 (d, J = 9.0 Hz, 2H).^{13}C{1H} NMR (126 MHz, DMSO) δ 166.82, 131.87, 131.47, 130.30, 127.01.

(4s) m-Floro benzoic acid:

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.91 (d, J = 9.2 Hz, 1H), 7.46 (td, J = 5.5, 8.0 Hz, 2H), 7.32 (td, J = 2.7, 8.2 Hz, 1H). ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 170.50, 163.69, 161.73, 131.55 (d, J = 7.4 Hz), 130.35 (d, J = 3.7 Hz), 126.10 (d, J = 3.7 Hz), 121.08 (d, J = 21.1 Hz), 117.20 (d, J = 23.0 Hz).

(4t) m-Chloro benzoic acid:

^{1}H NMR (500 MHz, DMSO) δ 13.26 (s, 1H), 7.92 (s, 1H), 7.70 (d, J = 8.0 Hz, 1H), 7.55 (d, J = 8.2 Hz, 1H). ^{13}C{^{1}H} NMR (126 MHz, DMSO) δ 166.10 (COOH), 133.35, 132.95, 132.73, 130.68, 128.84.

(4u) o-Bromo benzoic acid:

^{1}H NMR (500 MHz, DMSO) δ 7.72 (ddd, *J* = 1.8, 7.6, 11.4 Hz, 2H), 7.51 – 7.39 (m, 2H). ^{13}C{^{1}H} NMR (126 MHz, DMSO) δ 167.42 (COOH), 133.77, 132.52, 127.74, 119.94.

(4v) Furan-2-Carboxylic acid:

^{1}H NMR (500 MHz, DMSO) δ 8.74 (s, 1H), 7.63 (dd, *J* = 0.9, 1.8 Hz, 1H), 7.34 – 7.28 (m, 1H), 6.55 (dd, *J* = 1.8, 3.5 Hz, 1H). ^{13}C{1H} NMR (126 MHz, $CDCl_3$) δ 163.62, 147.68, 144.36, 120.29, 112.62.

(4w) Piperonylic acid:

^{1}H NMR (500 MHz, DMSO) δ 12.74 (s, 1H), 7.54 (dd, *J* = 1.8, 8.2 Hz, 1H), 7.36 (d, *J* = 1.8 Hz, 1H), 7.00 (d, *J* = 8.2 Hz, 1H), 6.12 (s, 2H). ^{13}C{1H} NMR (126 MHz, DMSO) δ 166.64, 151.15, 147.49, 124.98, 124.67, 108.80, 108.09, 101.95.

(4x) Butyric acid:

^{1}H ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.33 (t, *J* = 7.4 Hz, 2H), 1.66 (q, *J* = 7.5 Hz, 2H), 0.96 (d, *J* = 7.5 Hz, 3H). ^{13}C{1H} NMR (126 MHz, $CDCl_3$) δ 179.52, 35.77, 18.04, 13.48.

(4y) Hexan-1-ol:

No reaction

(4z) Phenylethan-1-ol:

No reaction

NMR Spectra of carboxylic acid products-

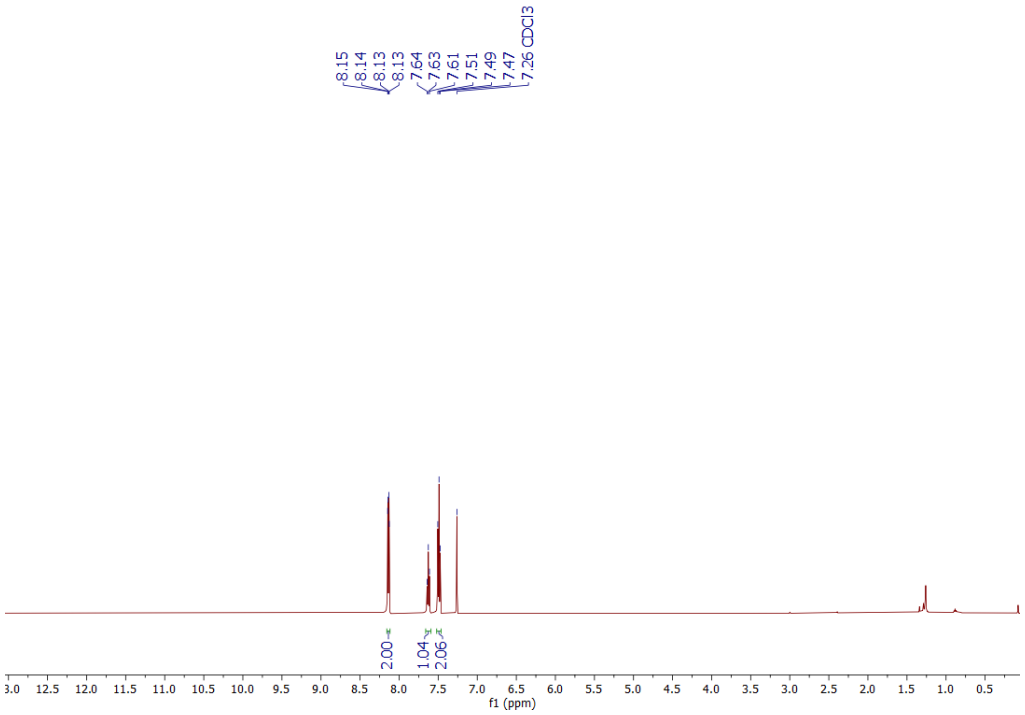


Figure S36. ^{1}H NMR of benzoic acid

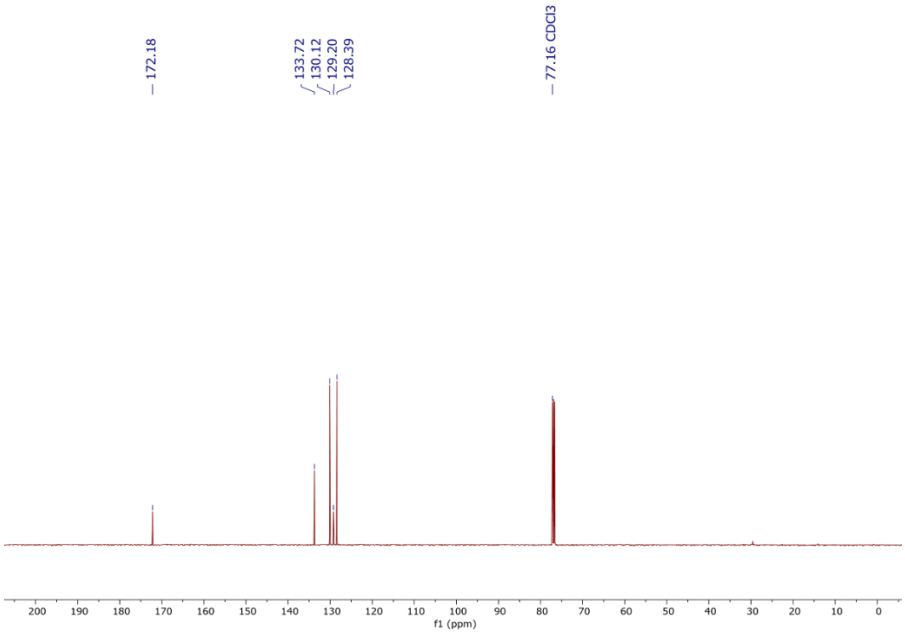


Figure S37. ^{13}C{^{1}H} NMR of benzoic acid

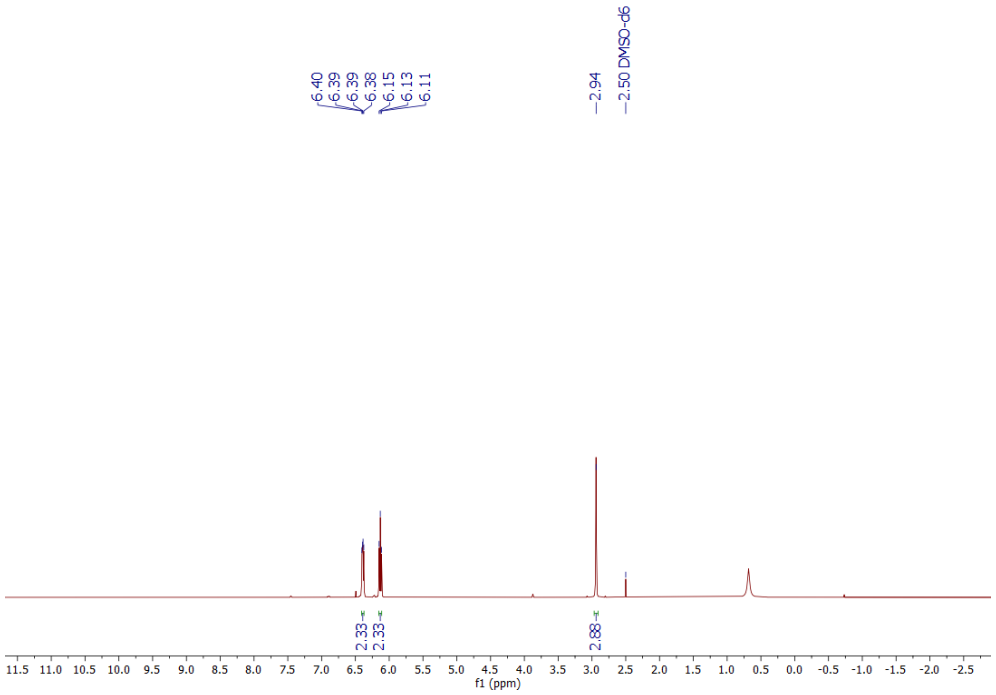


Figure S38. ^{1}H NMR of p-methoxy benzoic acid

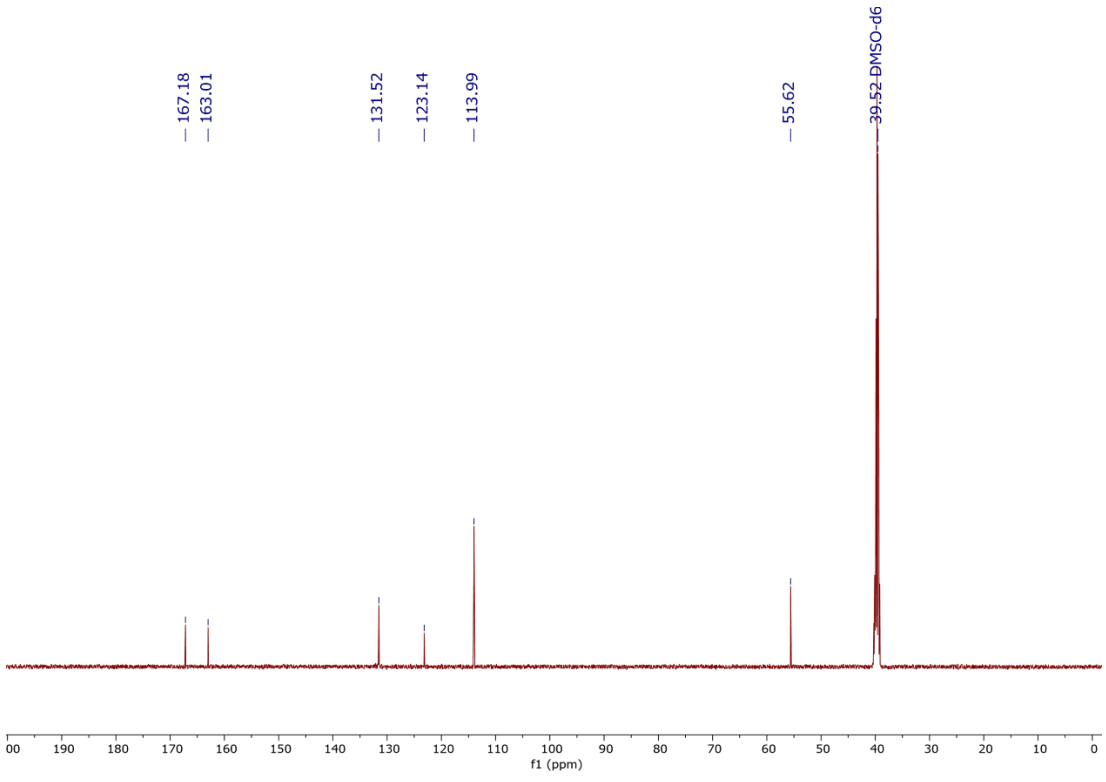


Figure S39. ^{13}C{^{1}H} NMR of p-methoxy benzoic acid

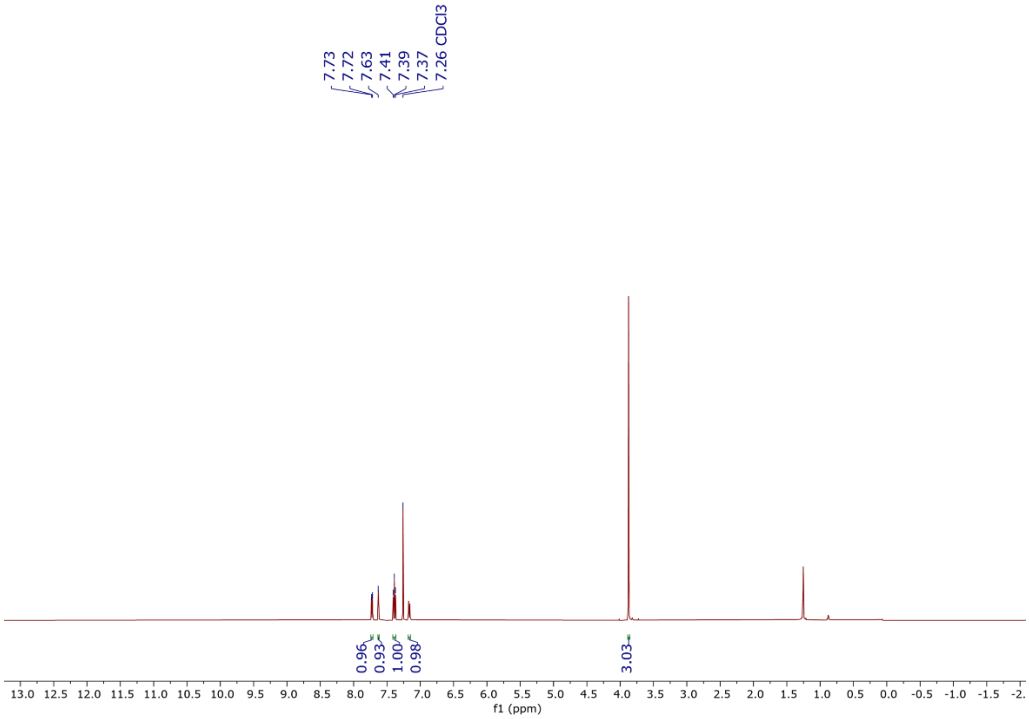


Figure S40. ^{1}HNMR of m-methoxy benzoic acid

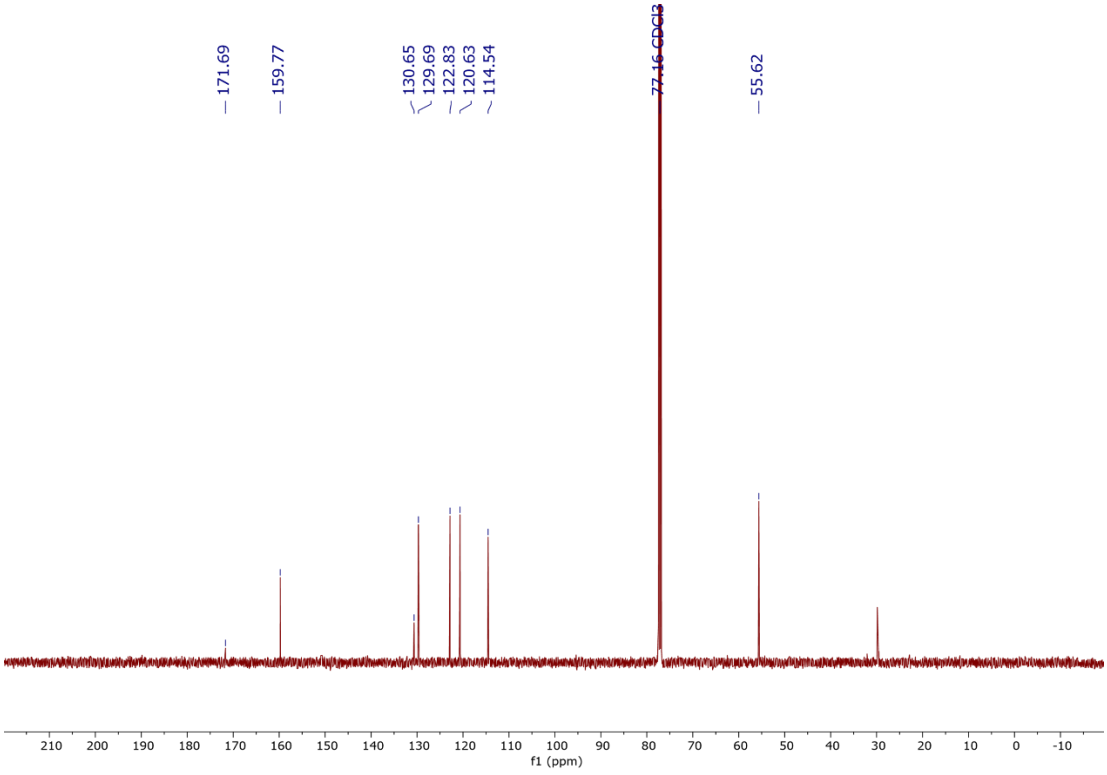


Figure S41. ^{13}C{^{1}H}NMR of m-methoxy benzoic acid

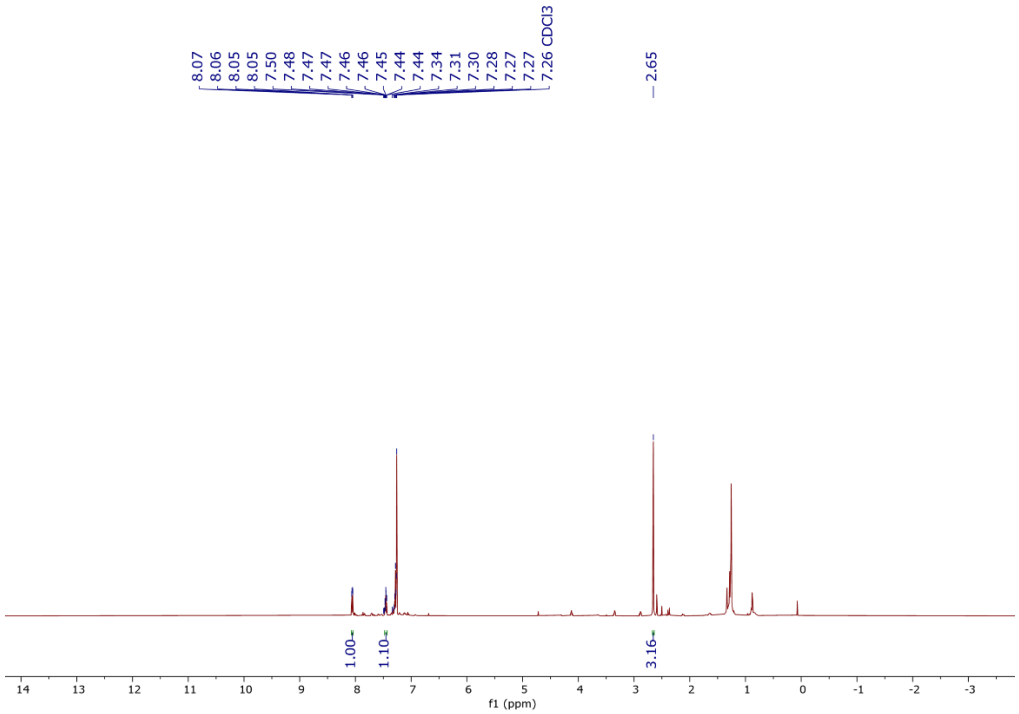


Figure S42. ^{1}H NMR of o-methoxy benzoic acid

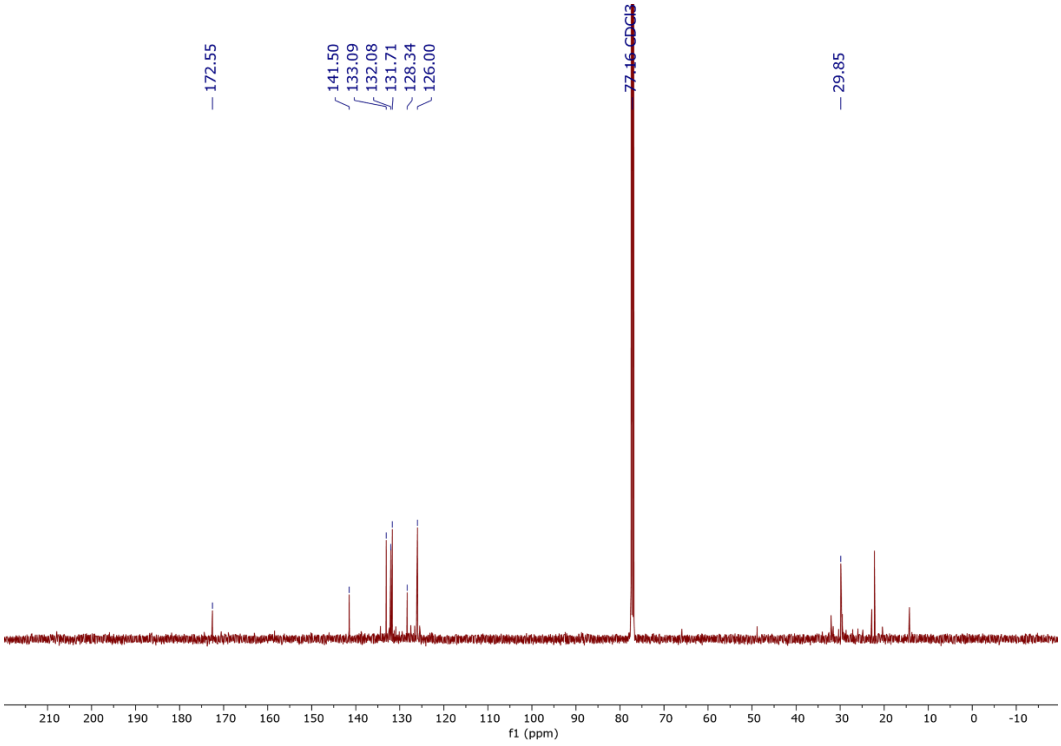


Figure S43. ^{13}C{^{1}H}NMR of o-methoxy benzoic acid

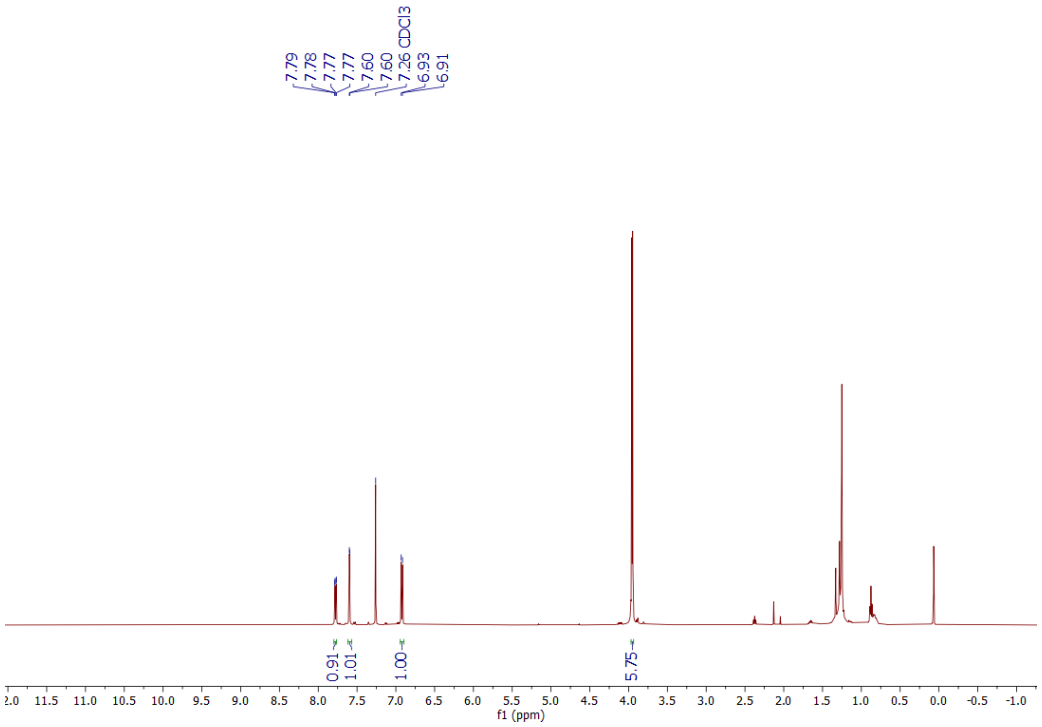


Figure S44. ^{1}H NMR of m,p-dimethoxy benzoic acid

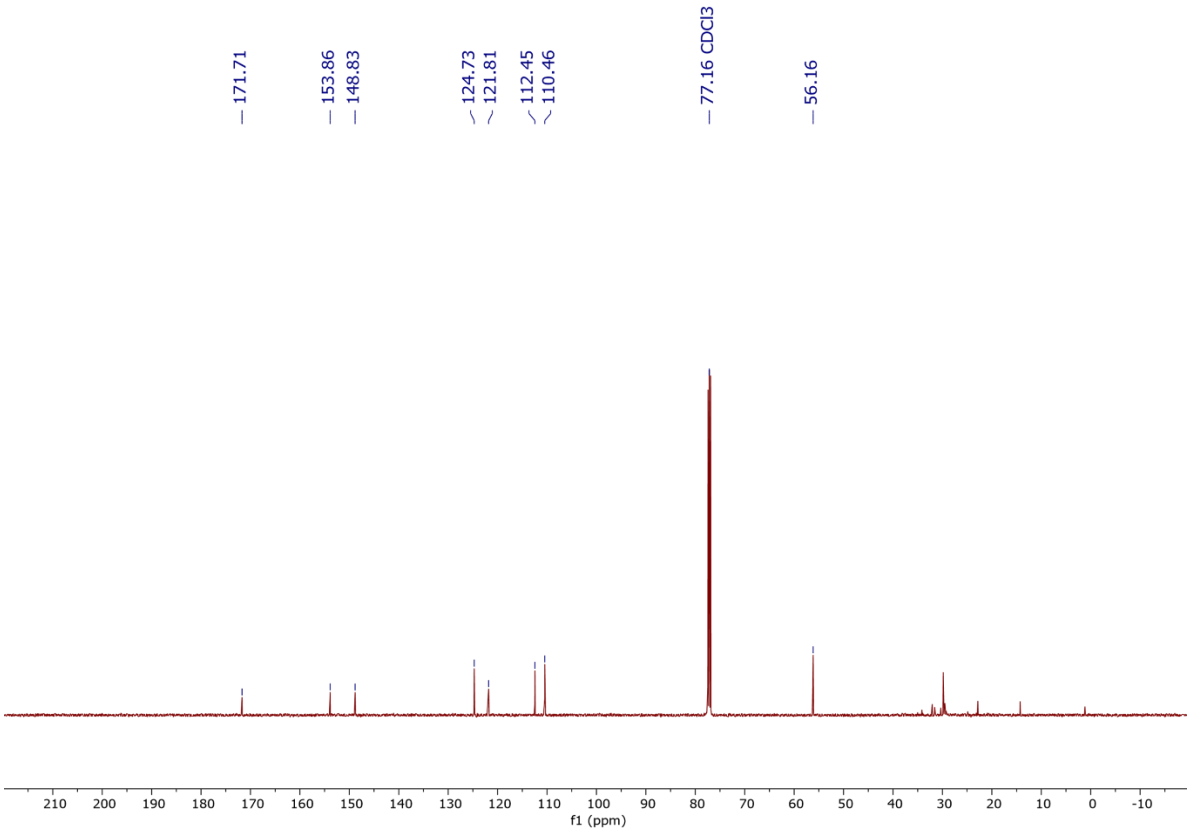


Figure S45. $^{13}C\{^{1}H\}$ NMR of m,p-dimethoxy benzoic acid

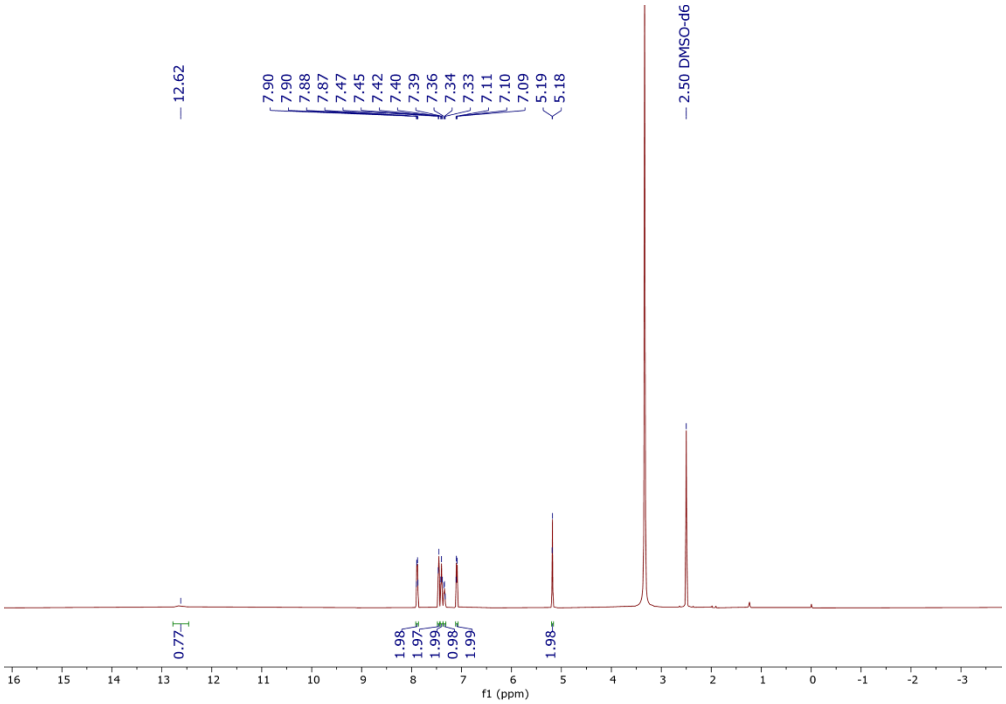


Figure S46. ^{1}H NMR of p-(benzyloxy)benzoic acid

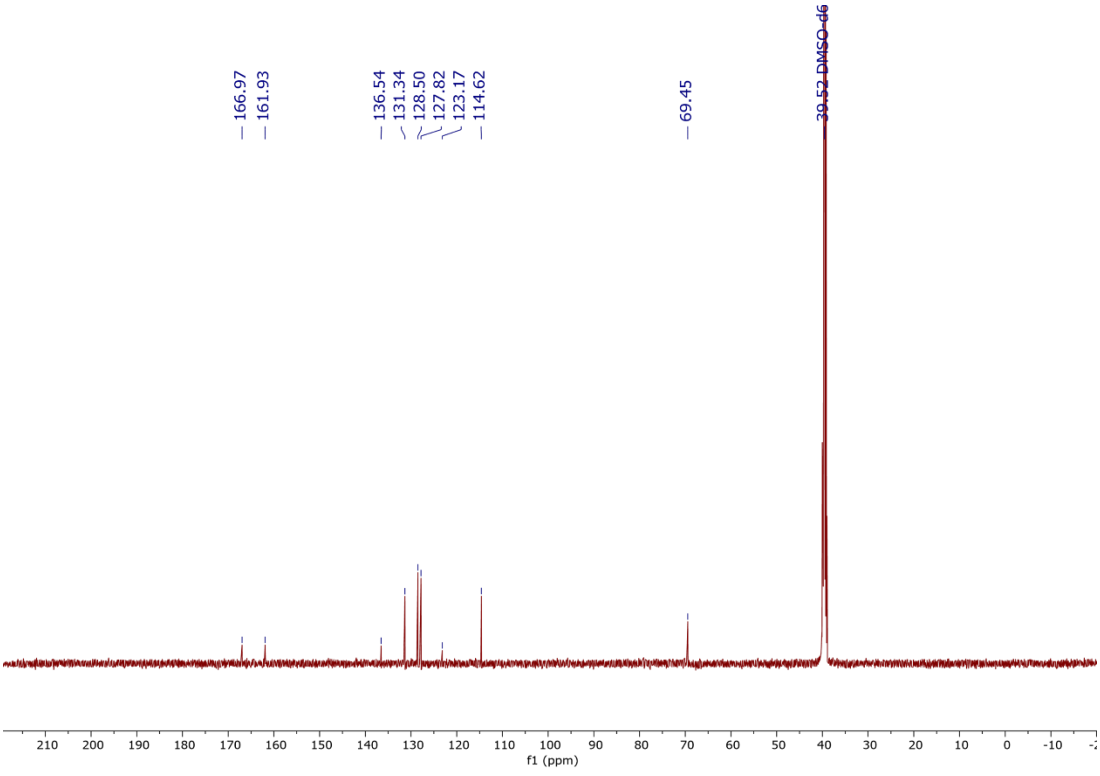


Figure S47. ^{13}C{^{1}H} NMR of p-(benzyloxy)benzoic acid

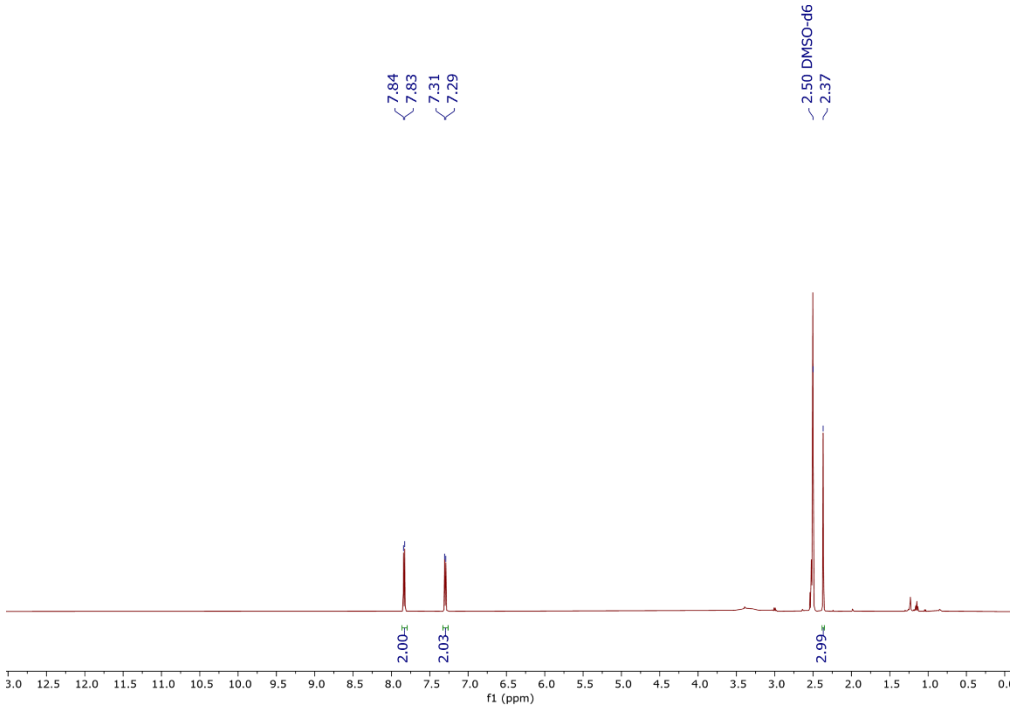


Figure S48. ^{1}H NMR of p-methyl benzoic acid

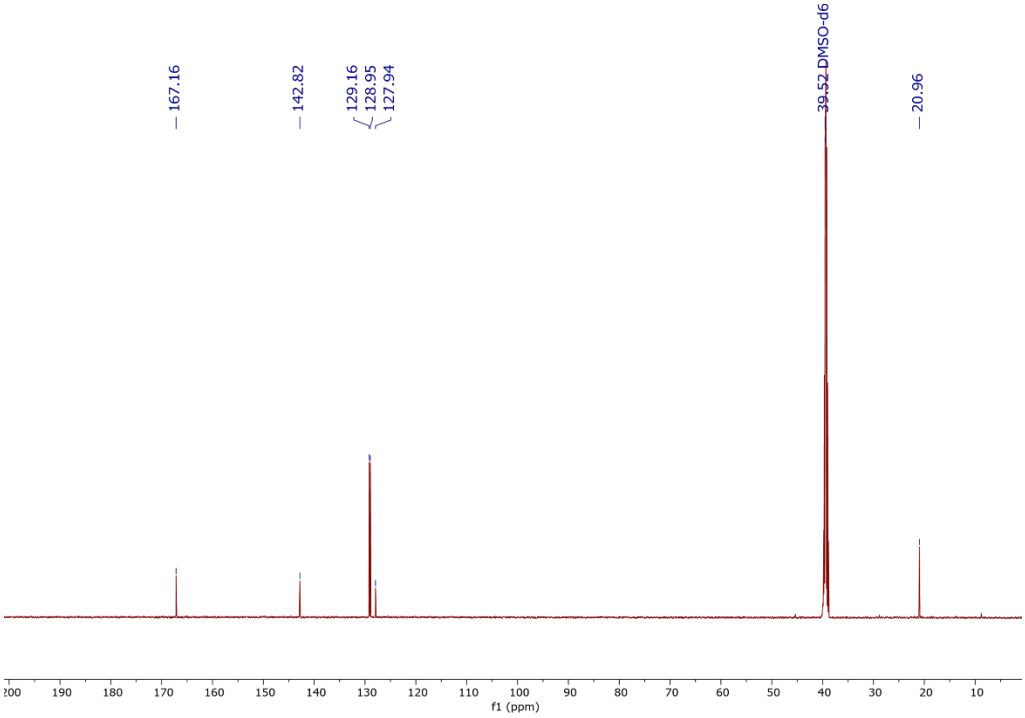


Figure S49. $^{13}C\{^{1}H\}$ NMR of p-methyl benzoic acid

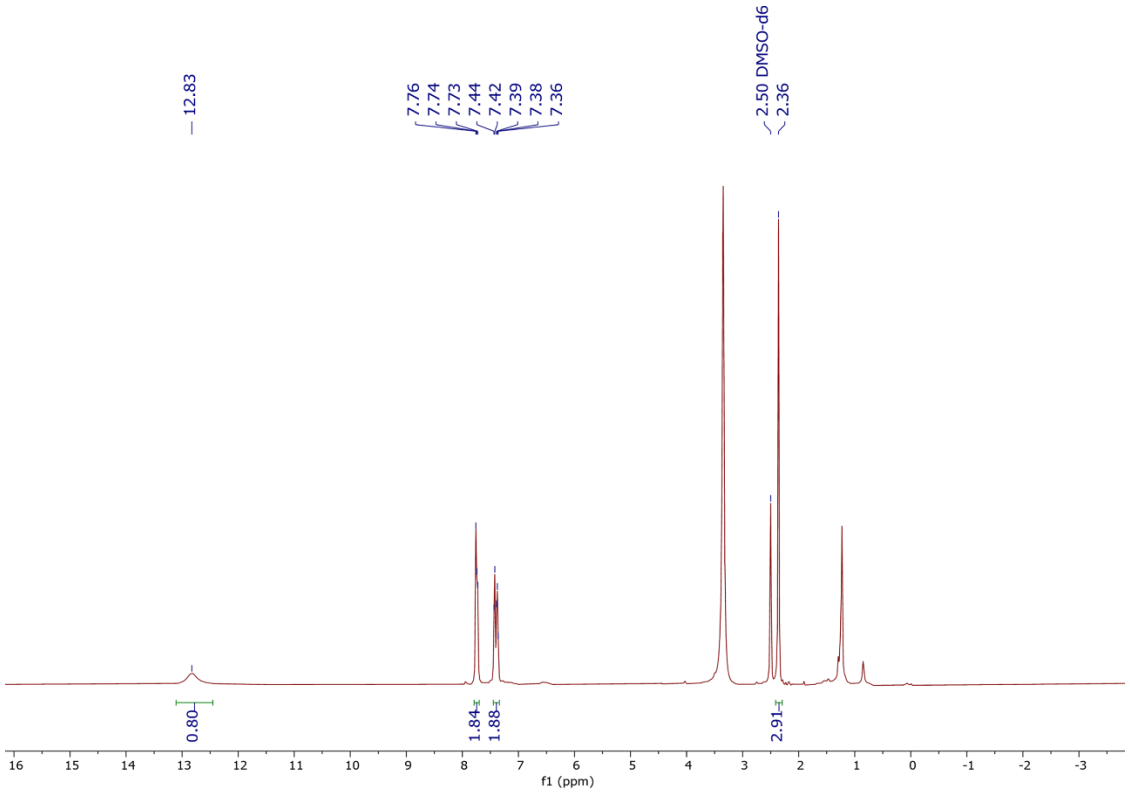


Figure S50. ^{1}H NMR of m-methyl benzoic acid

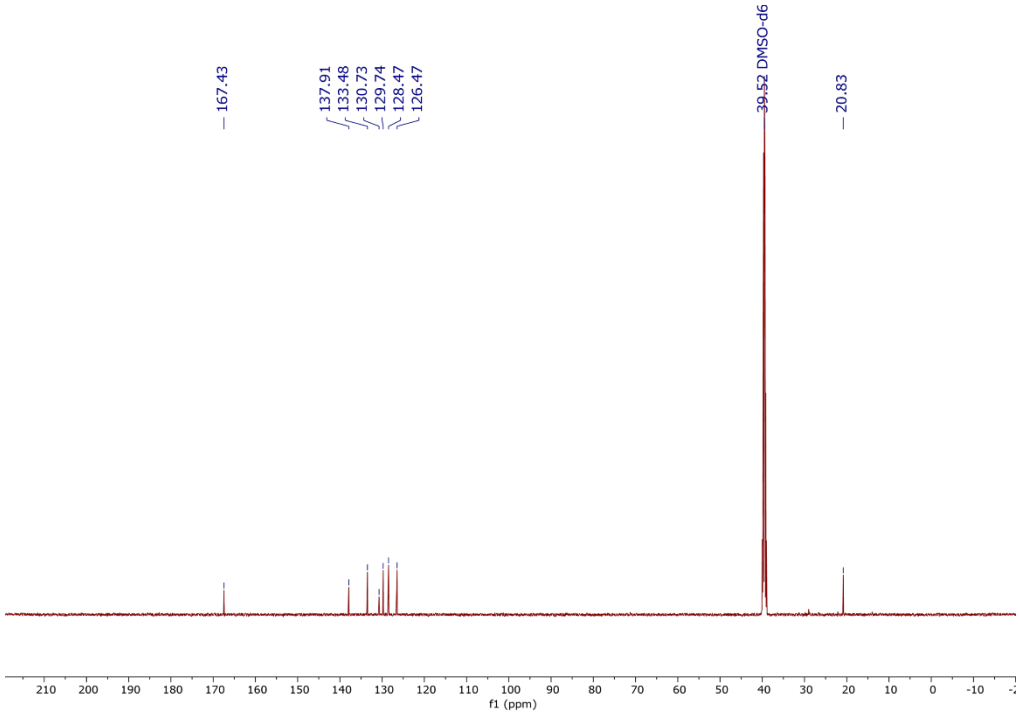


Figure S51. ^{13}C{^{1}H} NMR of m-methyl benzoic acid

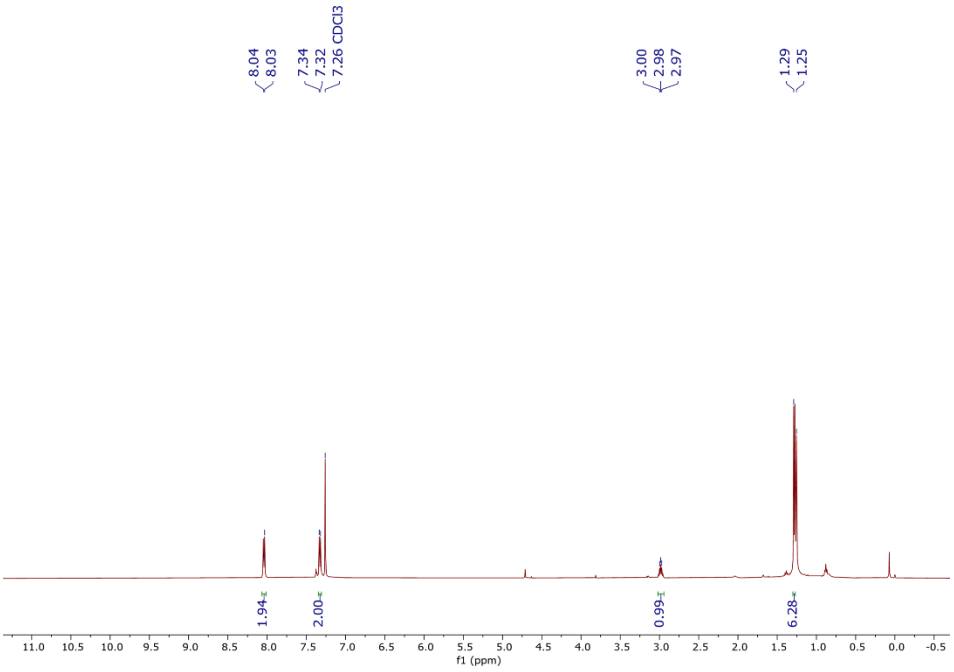


Figure S52. ^{1}H NMR of p-*iso*propyl benzoic acid

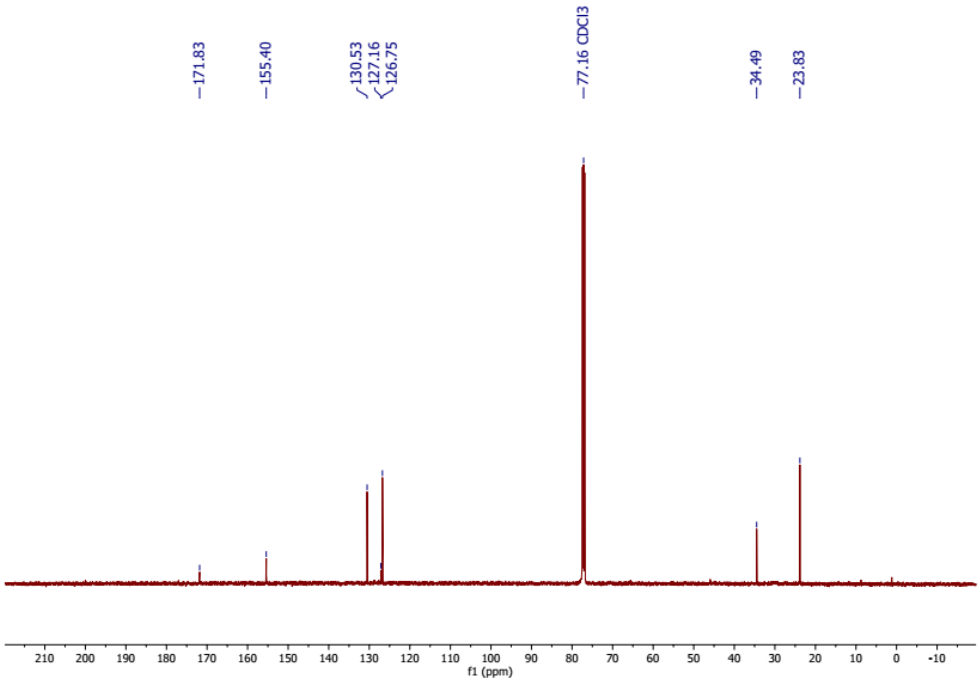


Figure S53. ^{13}C{^{1}H} NMR of p-*iso*propyl benzoic acid

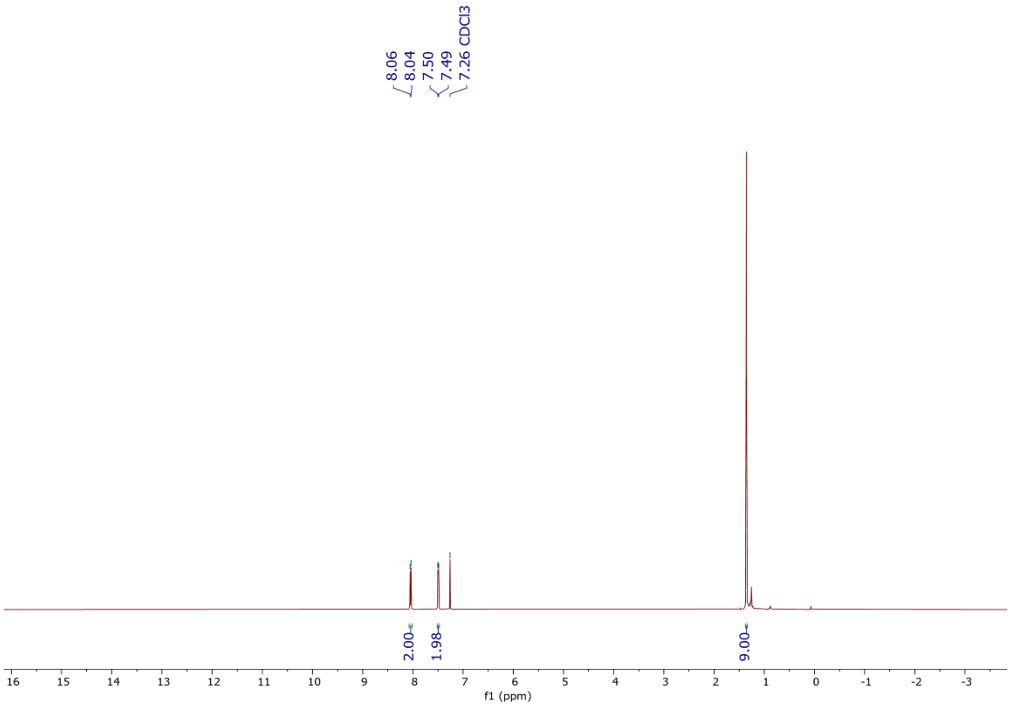


Figure S54. ^{1}H NMR of p-*tert*butyl benzoic acid

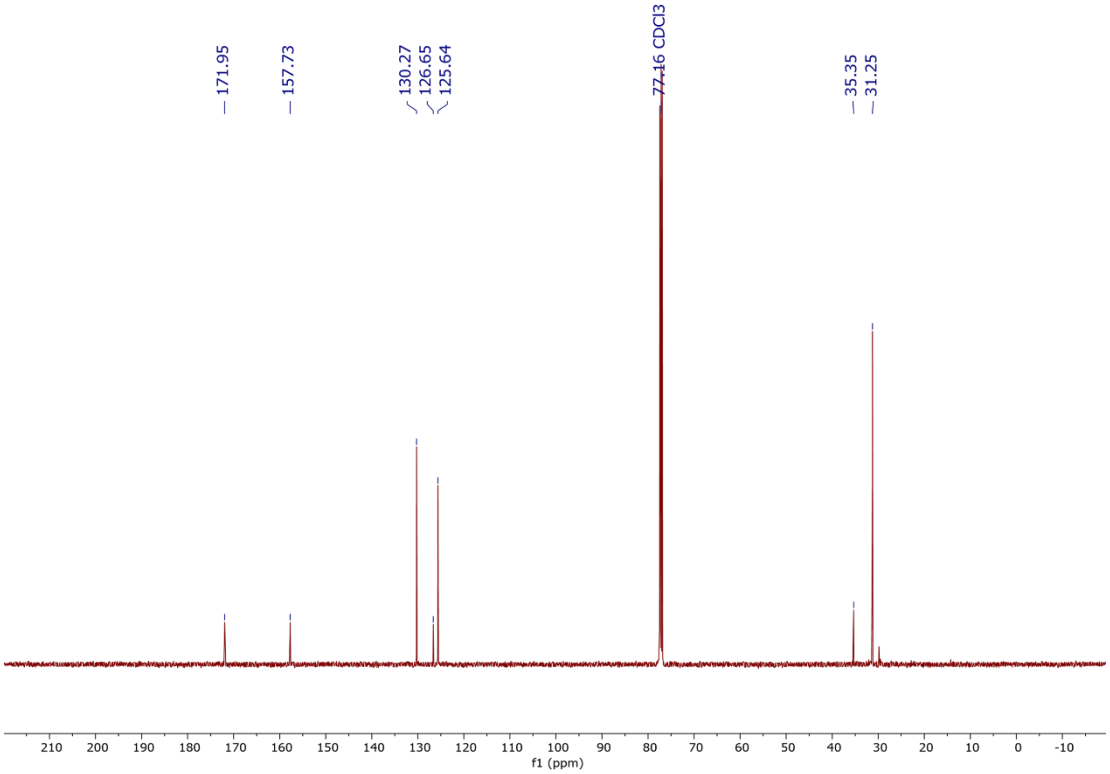


Figure S55. ^{13}C{^{1}H} NMR of p-*tert*butyl benzoic acid

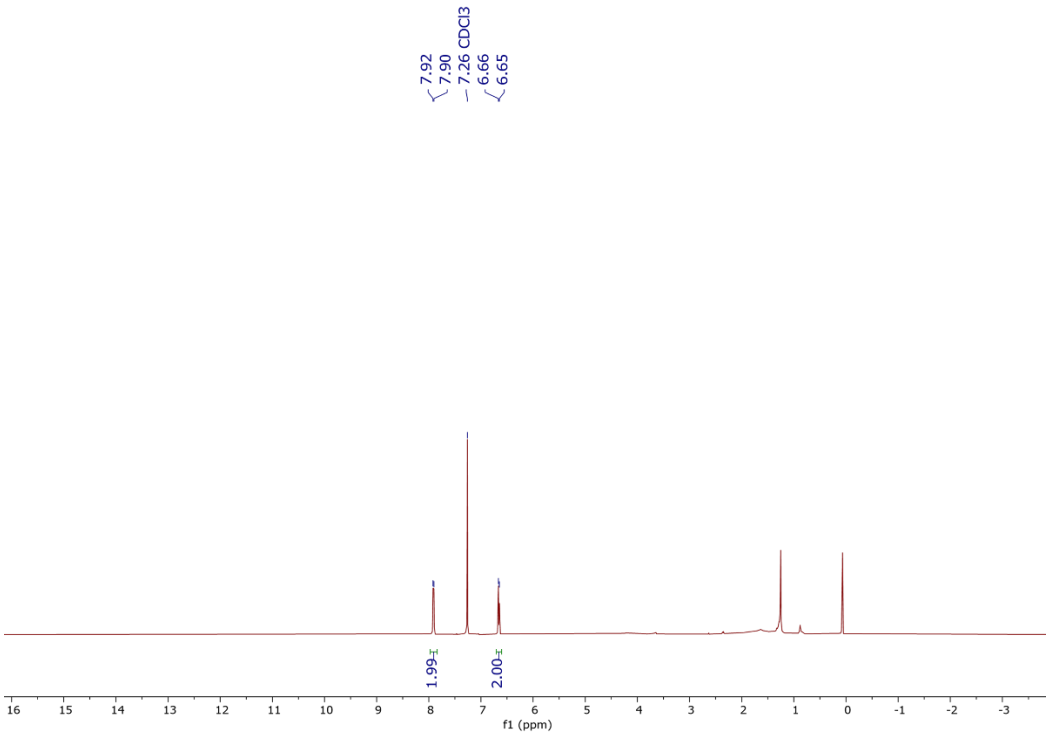


Figure S56. ^{1}H NMR of p-amino benzoic acid

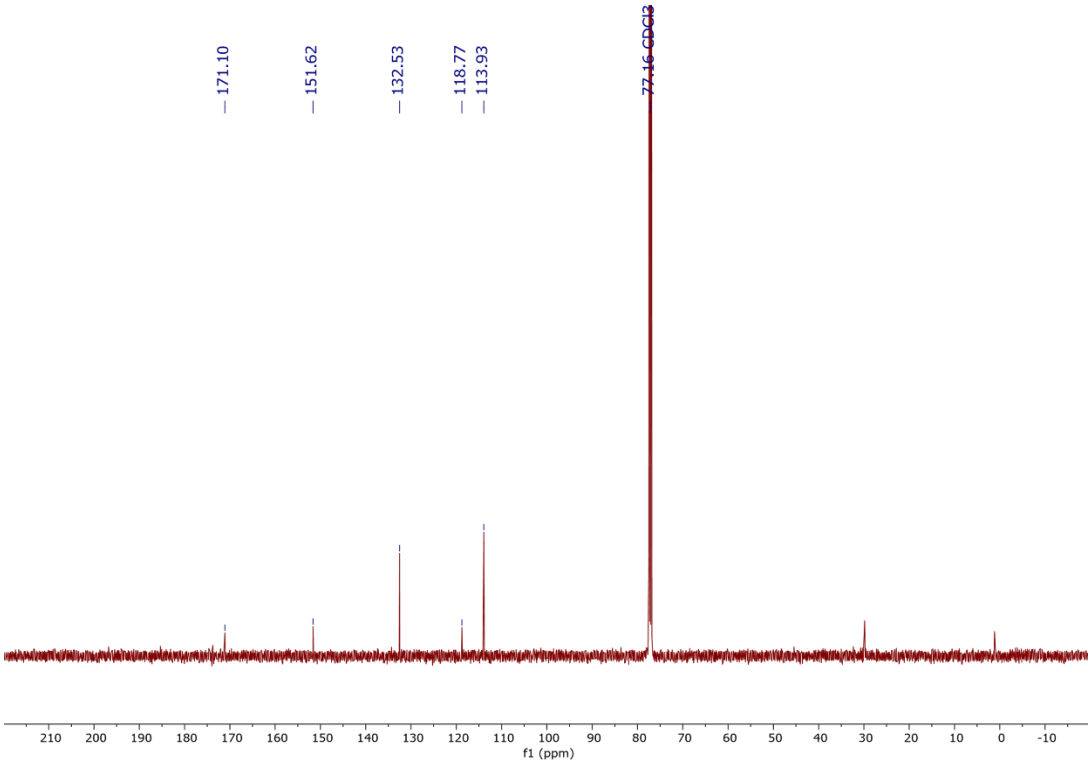


Figure S57. $^{13}C\{^{1}H\}$ NMR of p-amino benzoic acid

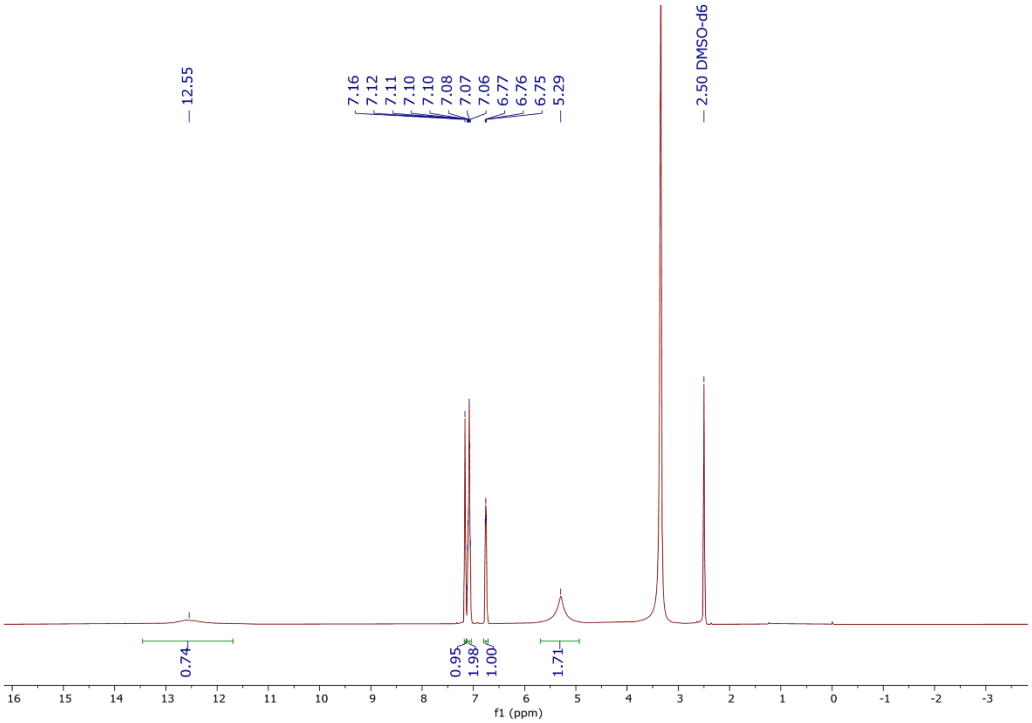


Figure S58. ^{1}H NMR of m-amino benzoic acid

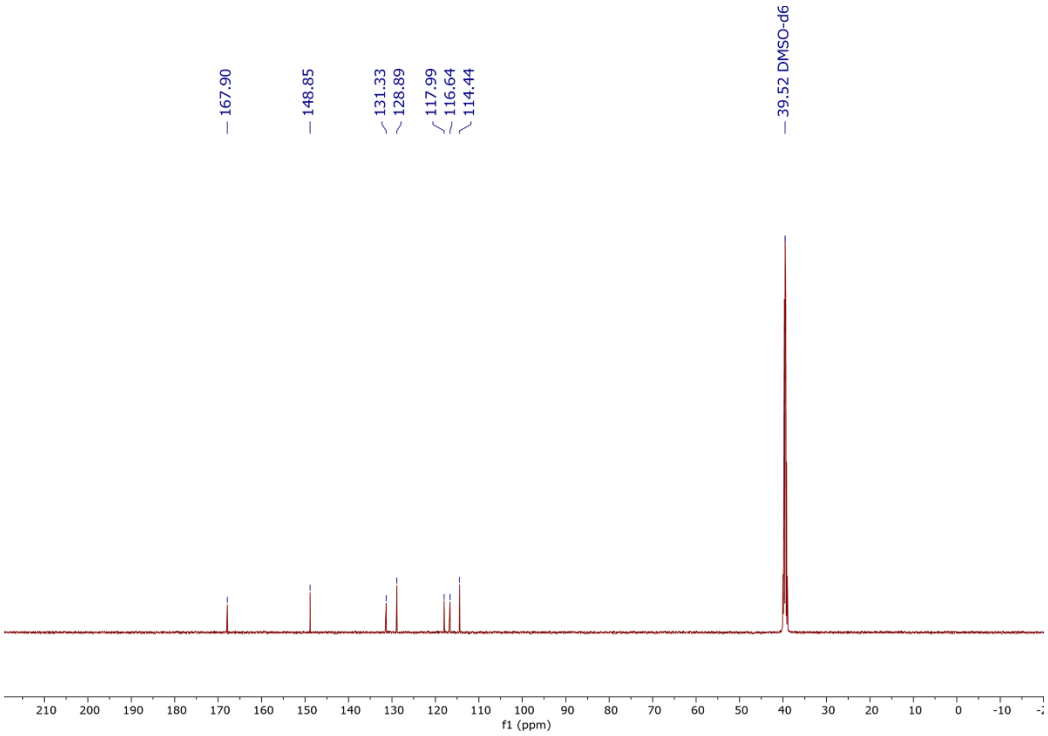


Figure S59. ^{13}C{^{1}H} NMR of m-amino benzoic acid

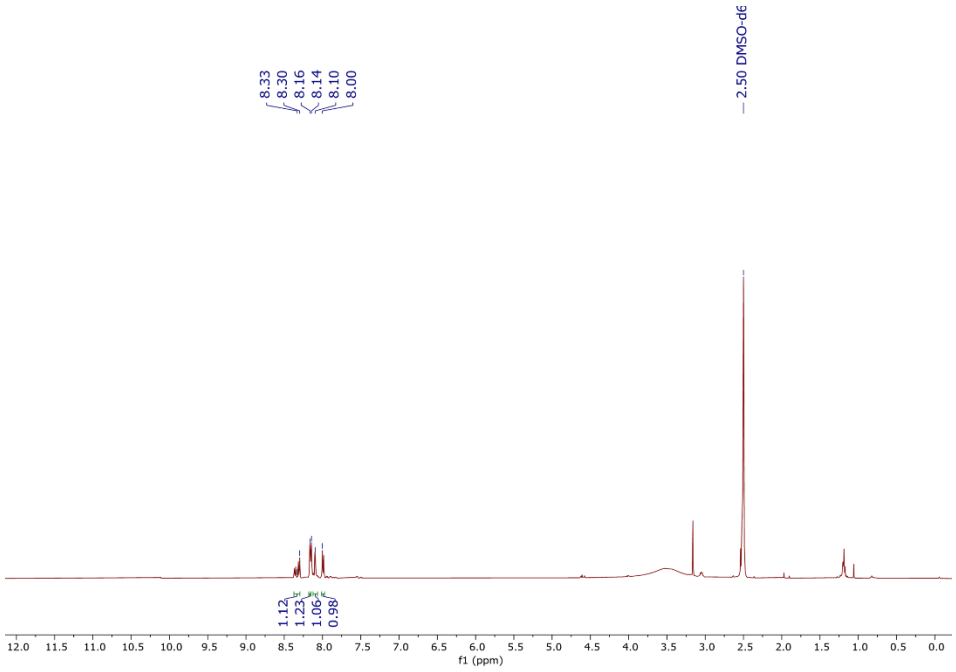


Figure S60. ^{1}H NMR of m-nitro benzoic acid

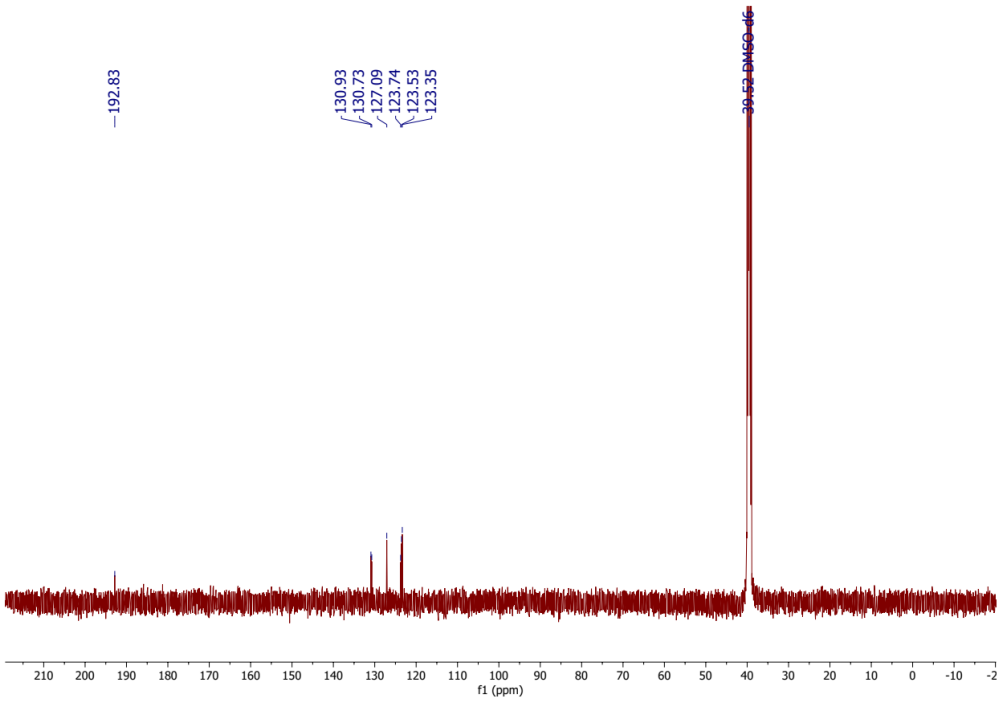


Figure S61. $^{13}C\{^{1}H\}$ NMR of m-nitro benzoic acid

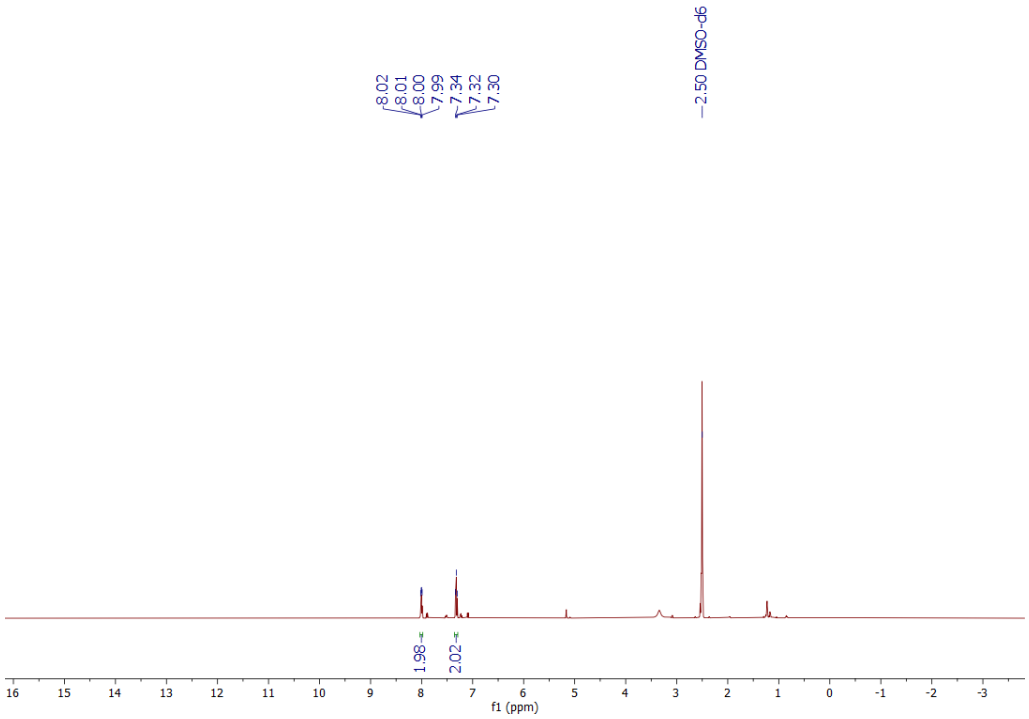


Figure S62. ^{1}H NMR of p-fluoro benzoic acid

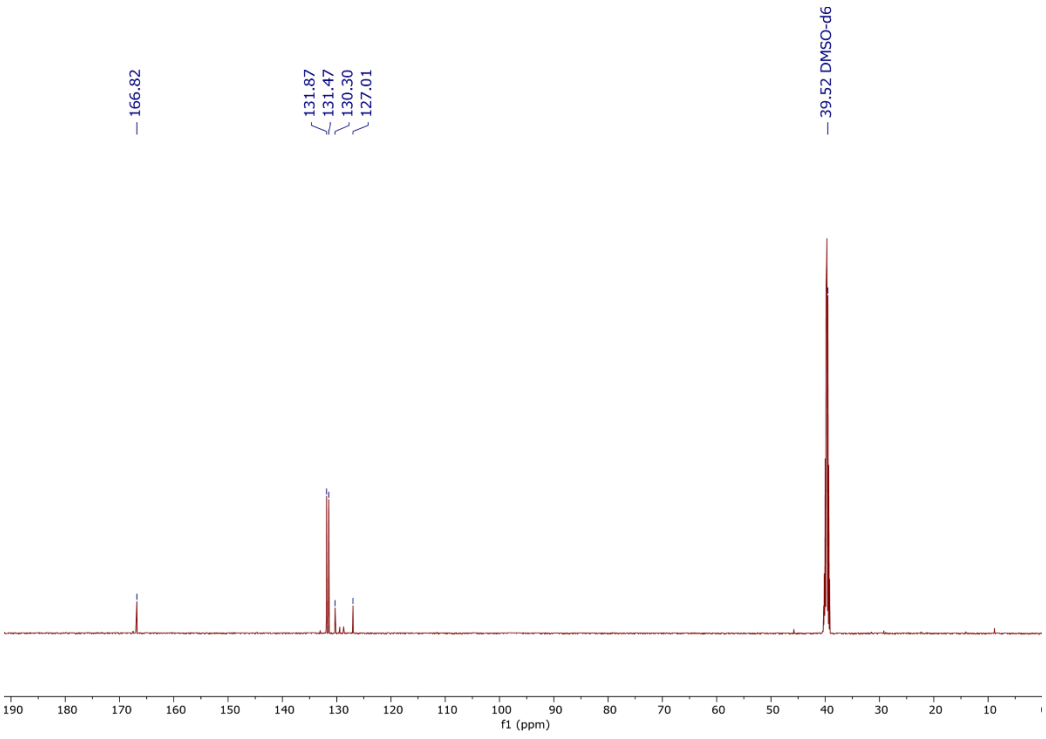


Figure S63. $^{13}C\{^{1}H\}$ NMR of p-fluoro benzoic acid

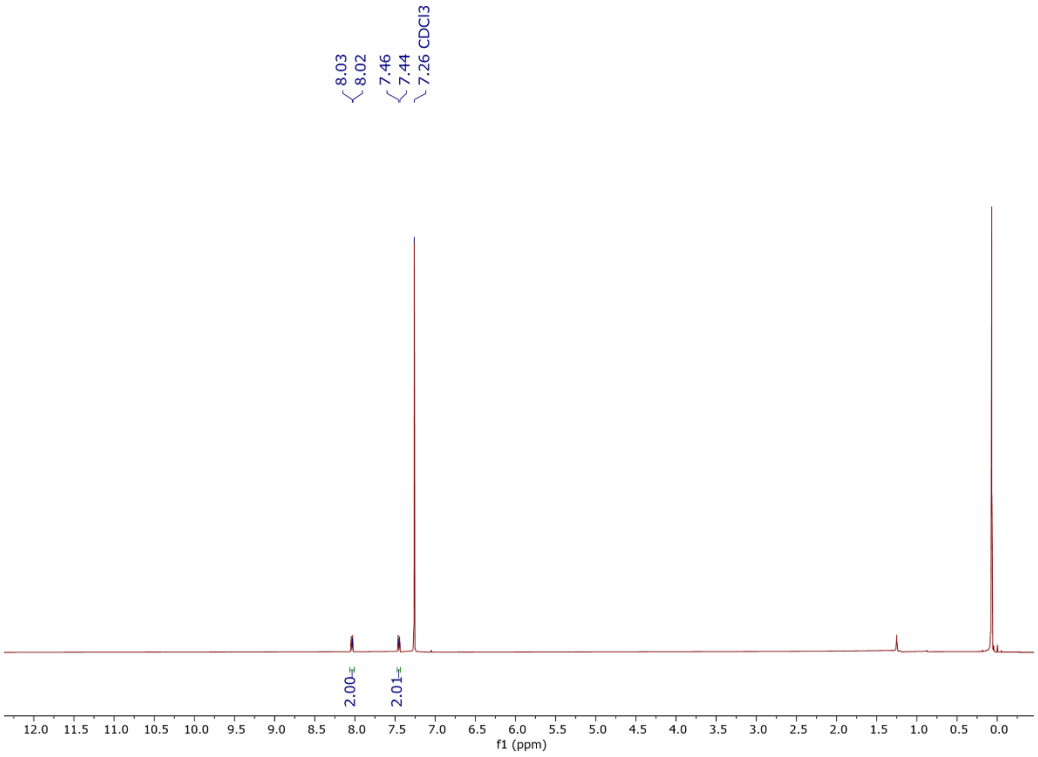


Figure S64. ^{1}H NMR of p-chloro benzoic acid

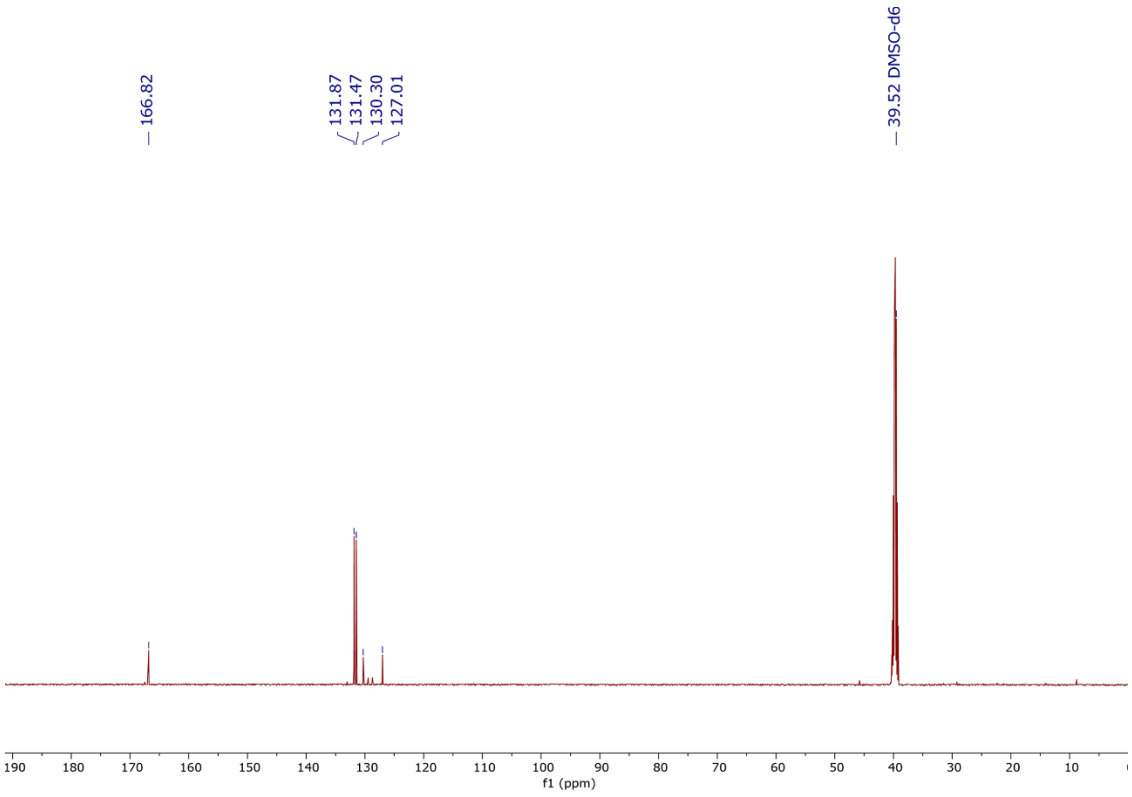


Figure S65. $^{13}C\{^{1}H\}$ NMR of p-chloro benzoic acid

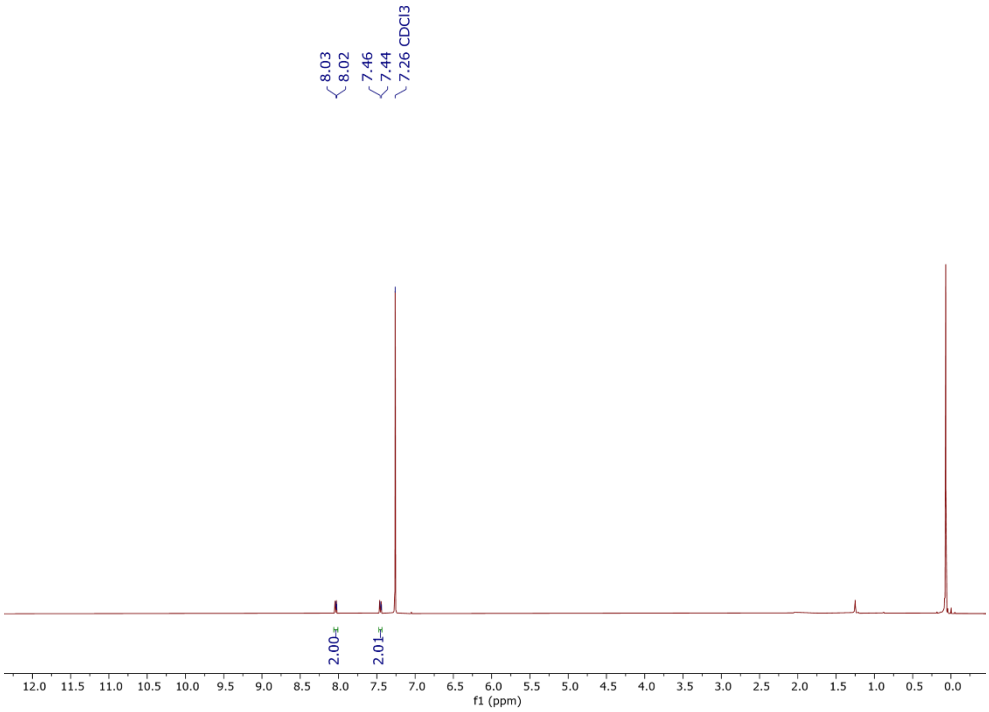


Figure S66. ^{1}H NMR of p-iodo benzoic acid

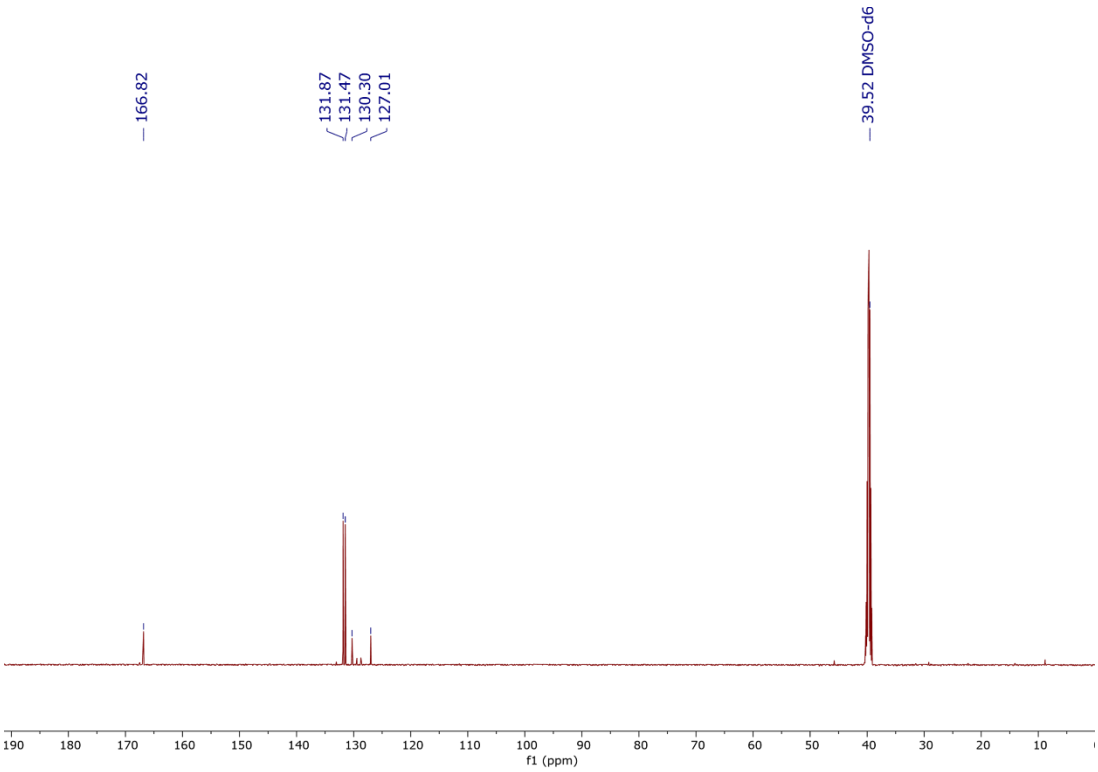


Figure S67. $^{13}C\{^{1}H\}$ NMR of p-iodo benzoic acid

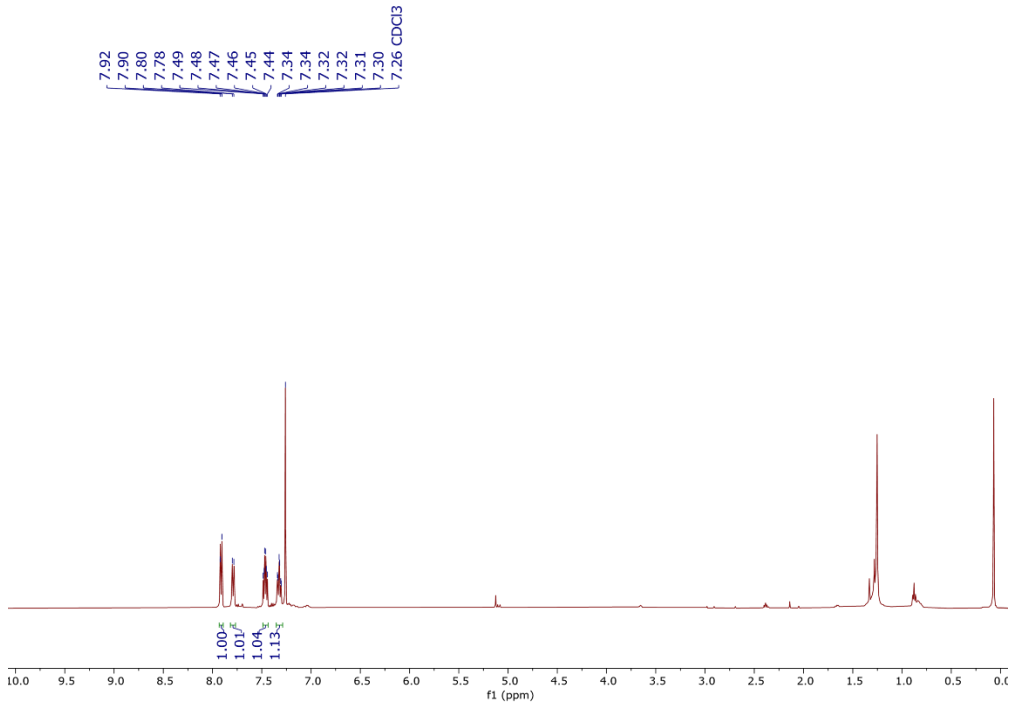


Figure S68. ^{1}H NMR of m-fluoro benzoic acid

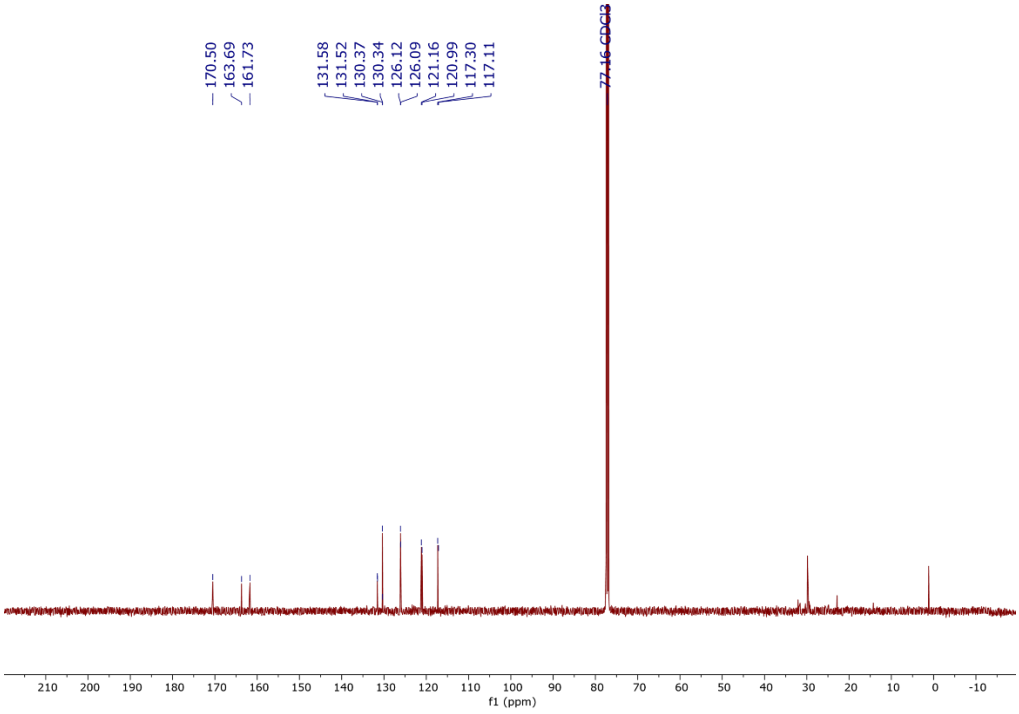


Figure S69. $^{13}C\{^{1}H\}$ NMR of m-fluoro benzoic acid

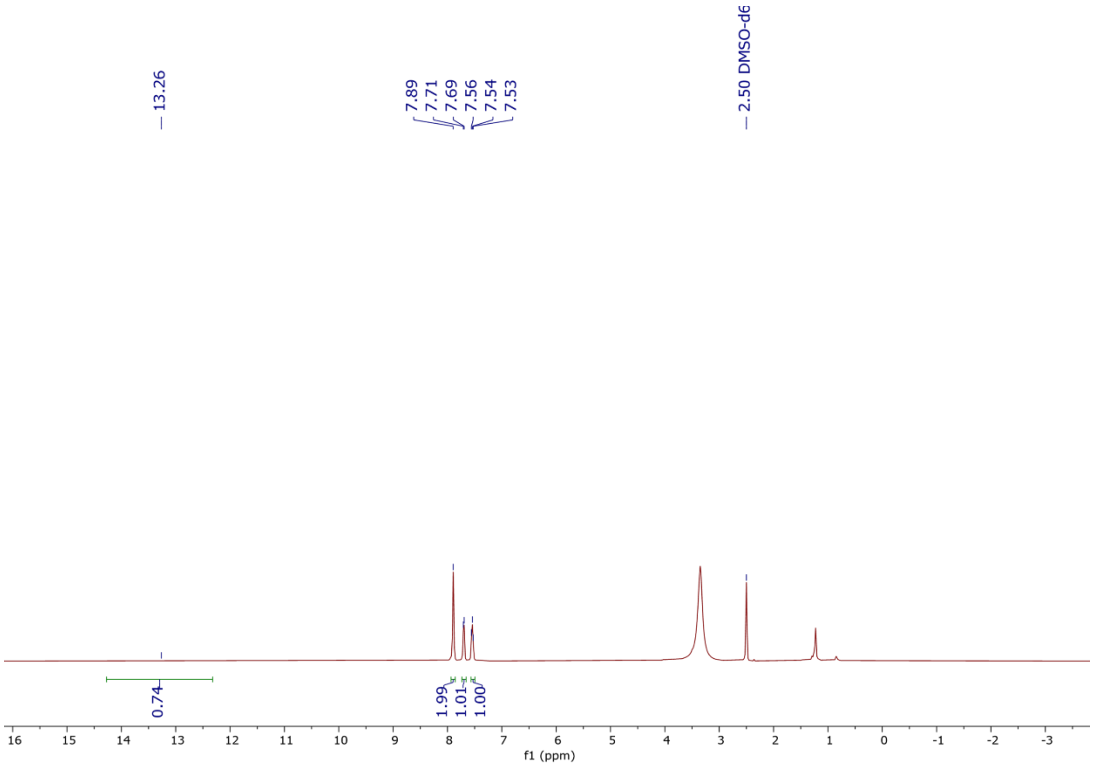


Figure S70. ^{1}H NMR of m-chloro benzoic acid

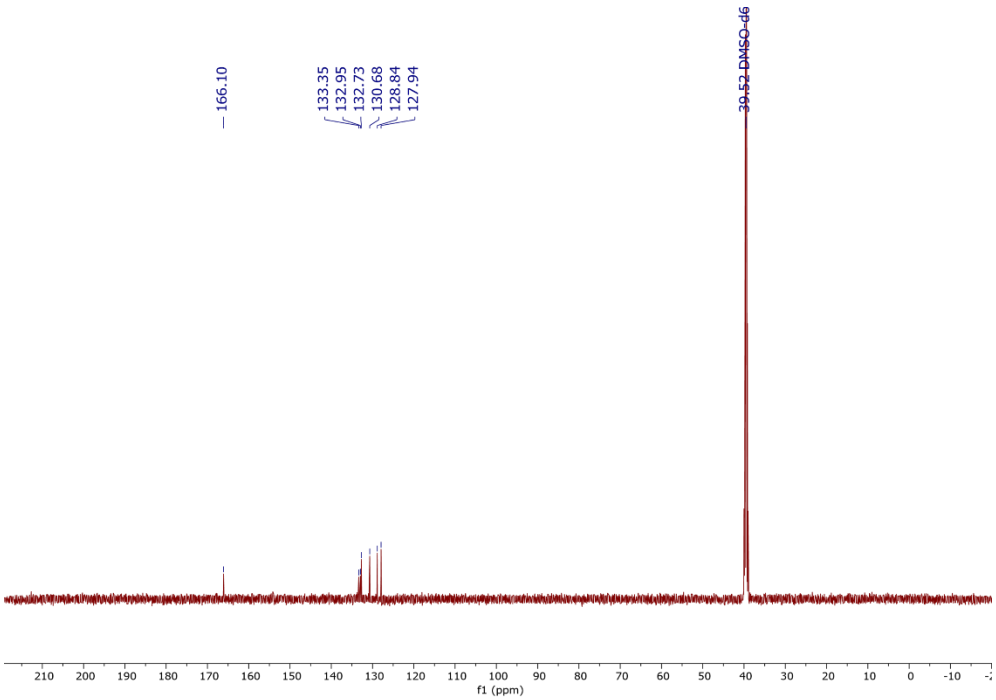


Figure S71. $^{13}C\{^{1}H\}$ NMR of m-chloro benzoic acid

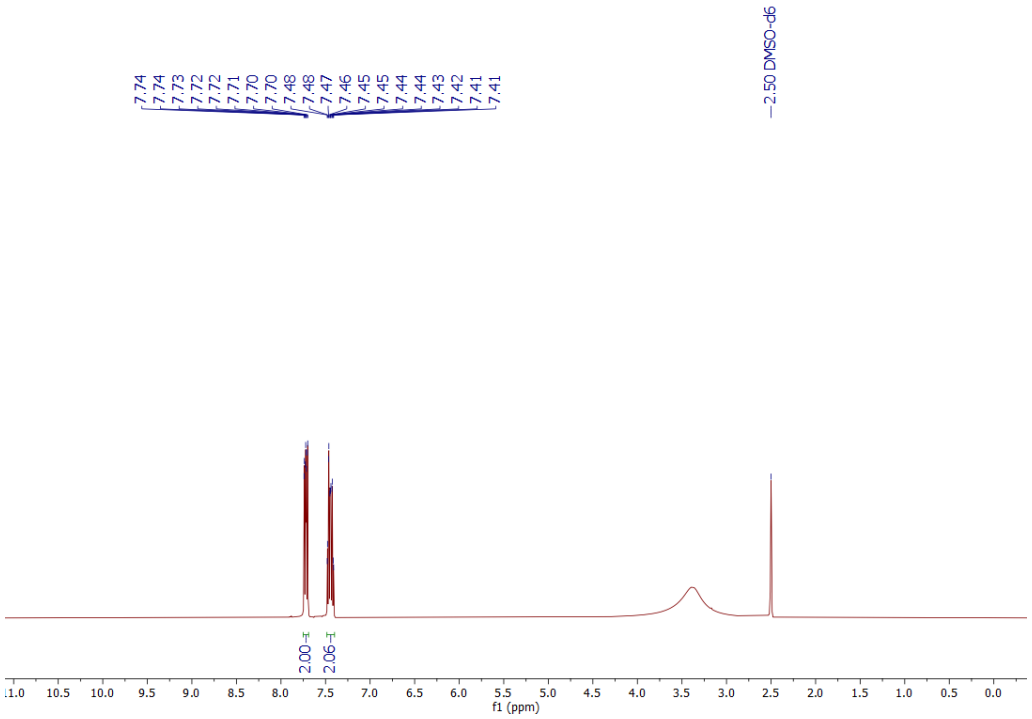


Figure S72. ^{1}H NMR of o-bromo benzoic acid

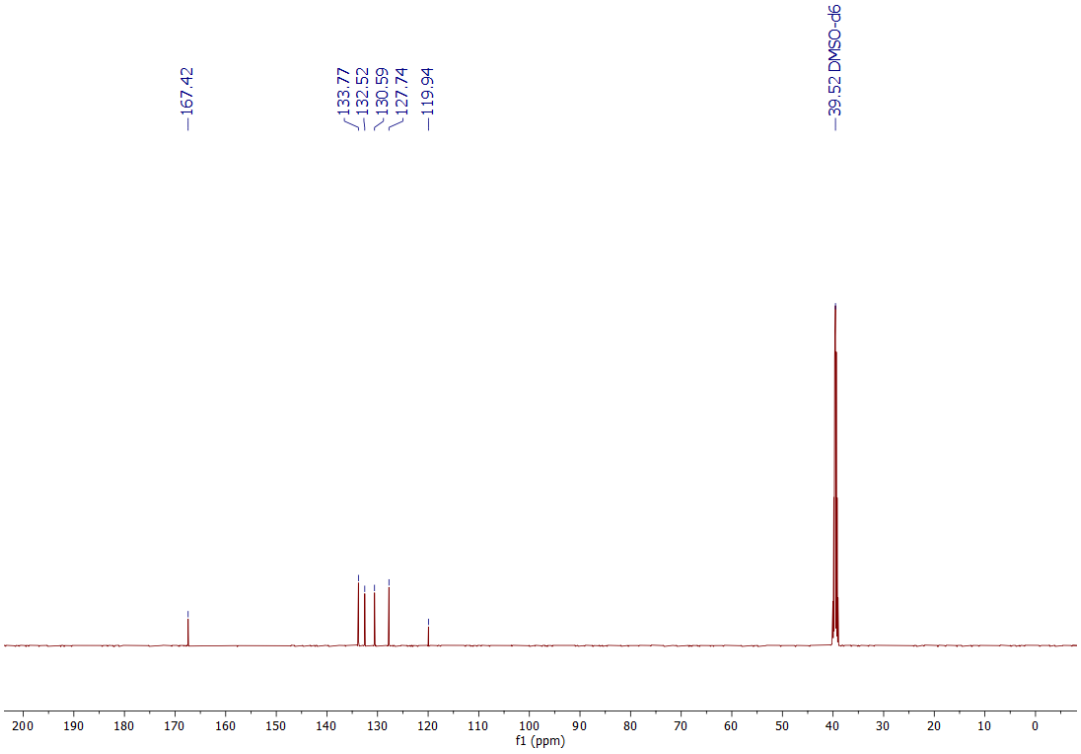


Figure S73. $^{13}C\{^{1}H\}$ NMR of o-bromo benzoic acid

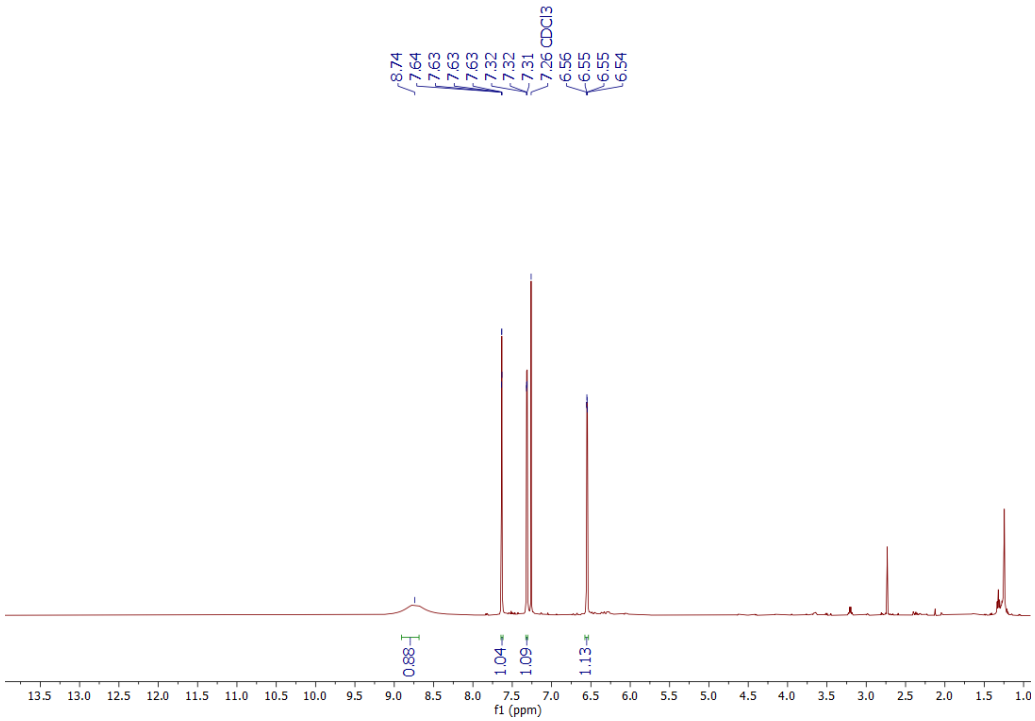


Figure S74. ^{1}H NMR of furan-2-carboxylic acid

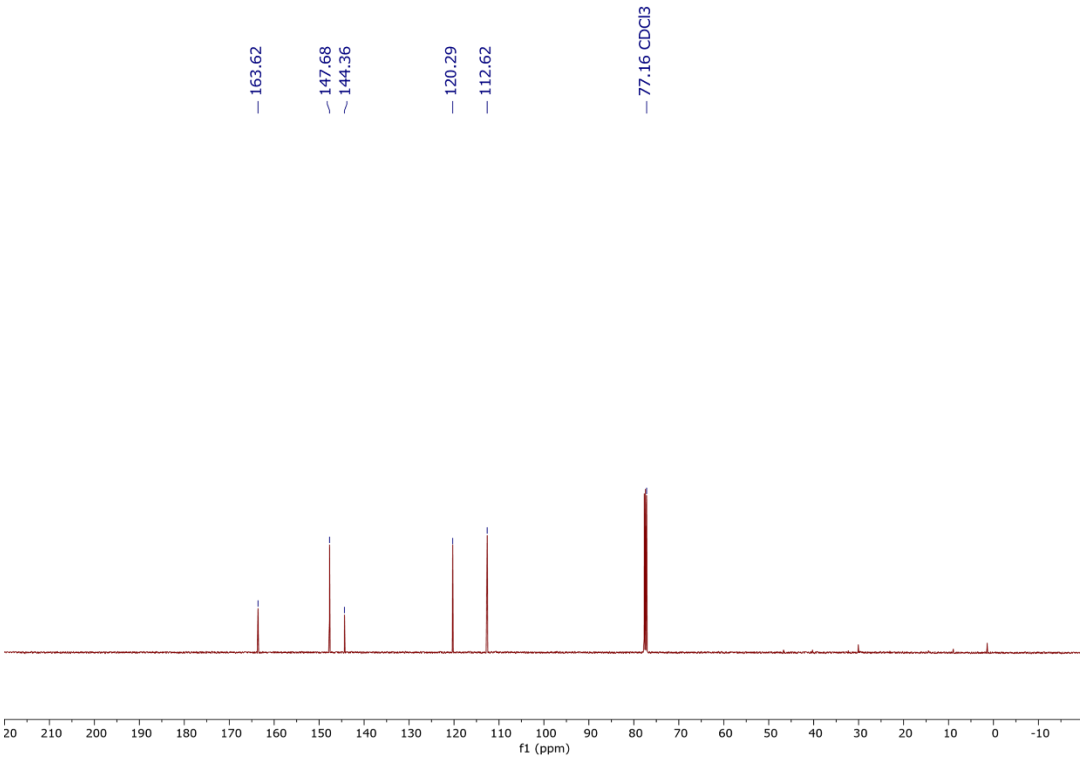


Figure S75. $^{13}C\{^{1}H\}$ NMR of furan-2-carboxylic acid

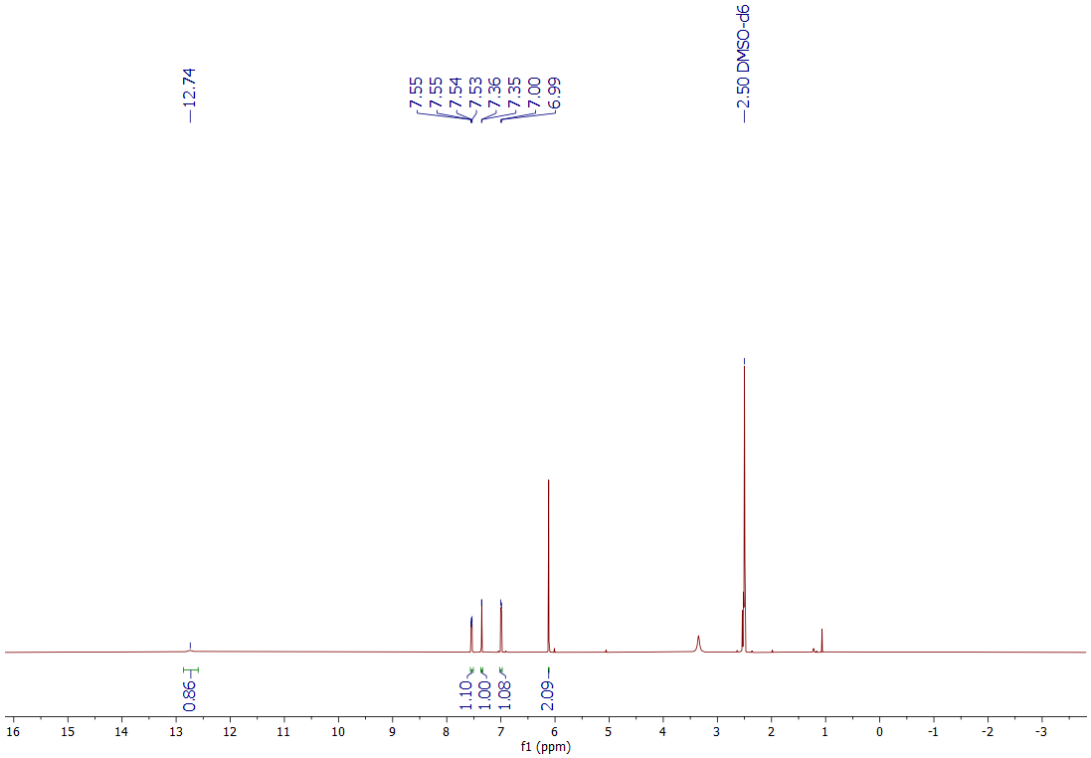


Figure S76. ^{1}H NMR of benzo[d][1,3]dioxole-5-carboxylic acid

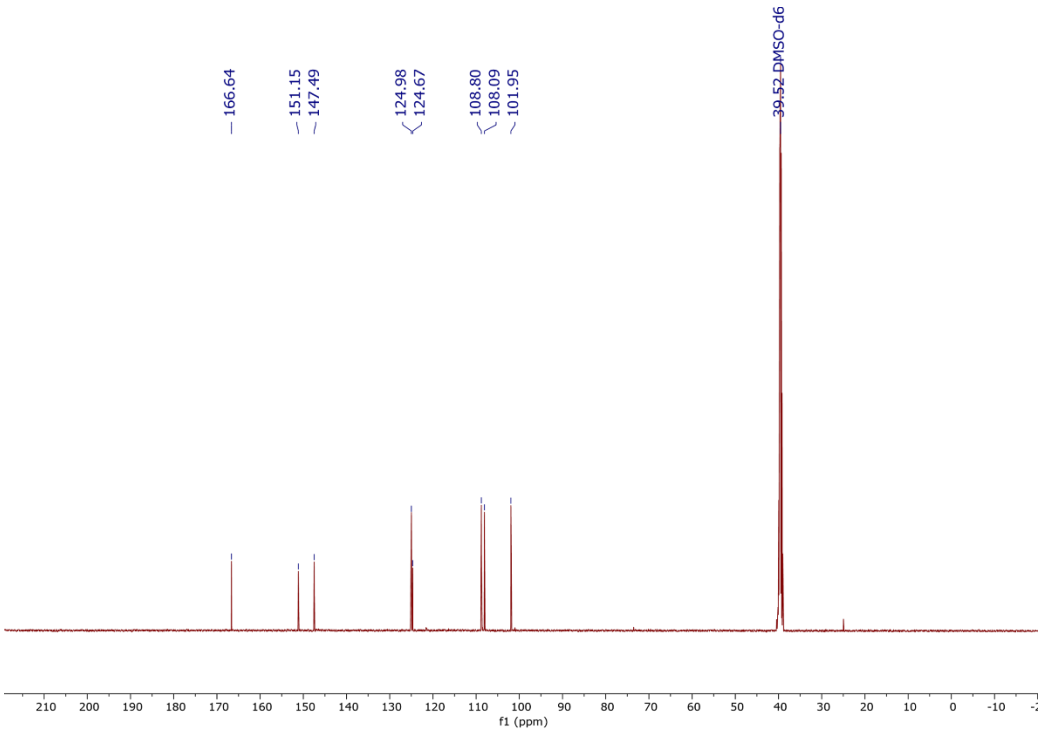


Figure S77. $^{13}C\{^{1}H\}$ NMR of benzo[d][1,3]dioxole-5-carboxylic acid

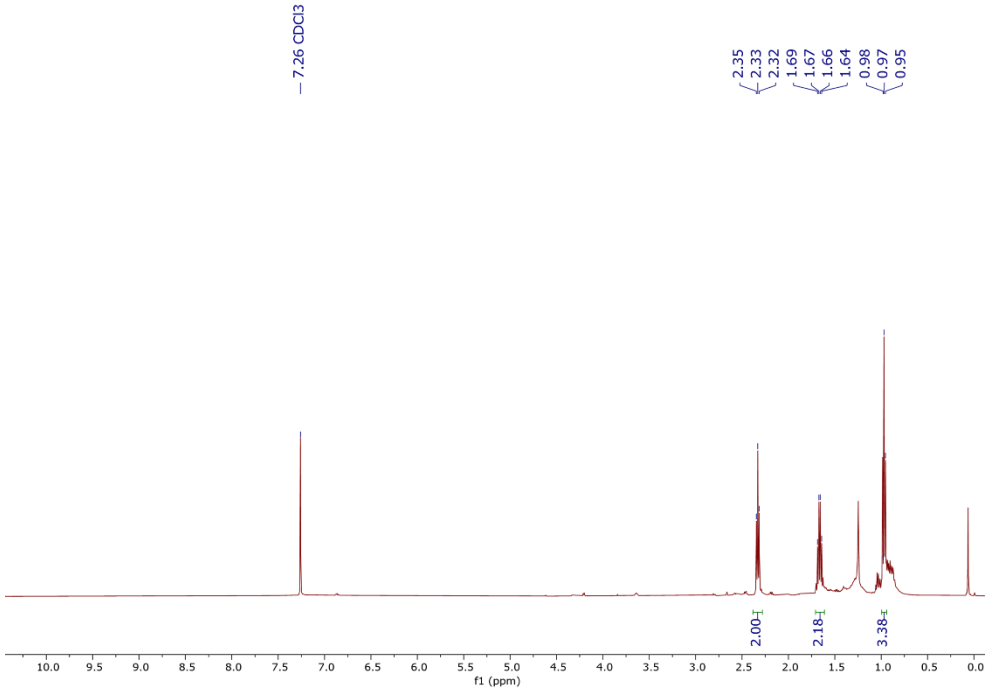


Figure S78. ^{1}H NMR of butyric acid

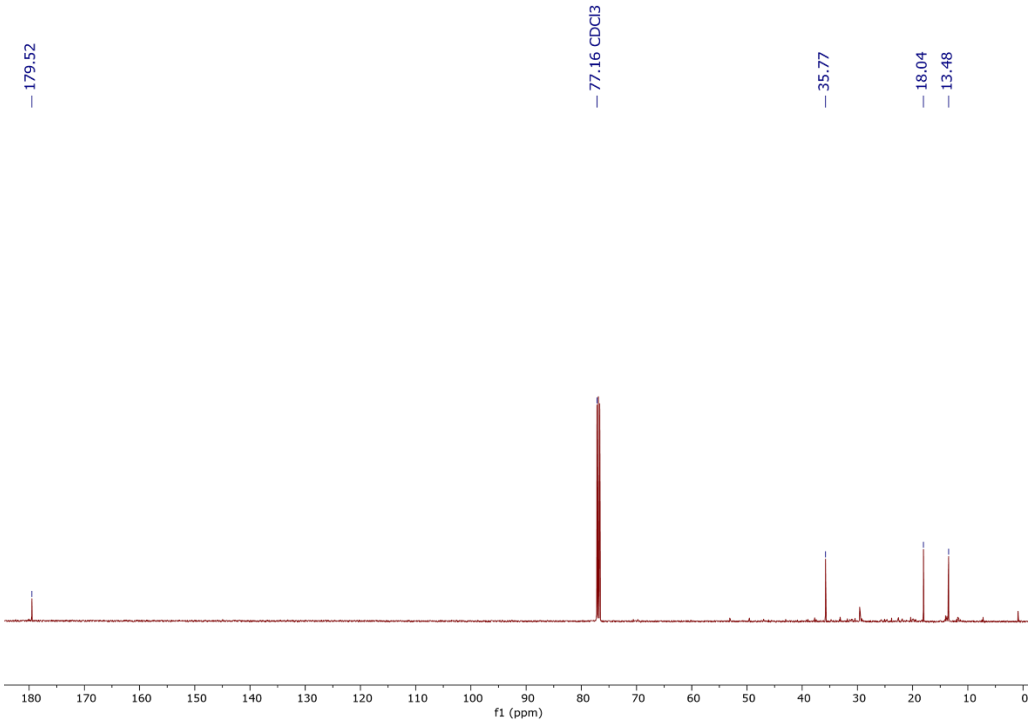


Figure S79. ^{13}C{^{1}H} NMR of butyric acid