Electronic Supporting Information for

Benzimidazolines convert sulphur dioxide to bisulfate at room temperature and atmospheric pressure utilizing aerial oxygen

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Experimental Section

Materials.

1,2-Phenylenediamine (SDFCL), p-toluenesulfonyl chloride (SDFCL), pyridine (SDFCL), dimethylsulfate (SDFCL), NaOH flakes (Fisher), 6-bromopyridine-2-carboxaldehyde (TCI India), benzaldehyde (Rankem) and salicylaldehyde (SRL) were used as received from commercial sources. Solvents were distilled under dry nitrogen atmosphere using conventional methods. *N*,*N*'-(1,2-Phenylene)bis(*N*,4-dimethylbenzenesulfonamide),[1] N^1,N^2-dimethylbenzene-1,2-diamine[2] and 1,3-dimethyl-2-phenyl-2,3-dihydro-1H-benzo[d]imidazole[3] were synthesized by following literature methods.

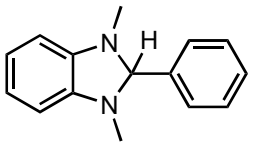
Methods.

NMR spectra were recorded on JEOL 500 MHz and JEOL 400 MHz spectrometers. Temperature was kept constant using a variable temperature unit within the error limit of ±1 K. The software MestReNova[4] was used for the processing of the NMR spectra. Tetramethylsilane (TMS) or the deuterated solvent residual peaks were used for calibration. Mass spectrometry experiments were performed on a Waters-Q-ToF-Premier-HAB213 equipped with an electrospray interface. Spectra were collected by constant infusion of the sample dissolved in methanol or acetonitrile with 0.1% formic acid. The freeware mMass was used to simulate the calculated isotopic distributions.[5]

Crystal Structure Determinations.

Single-crystal X-ray data were collected at 123 K on a Bruker SMART APEX CCD diffractometer using graphite-monochromated Mo Kα radiation (λ = 0.71069 Å). The linear absorption coefficients, the scattering factors for the atoms, and the anomalous dispersion corrections were taken from International Tables for X-ray Crystallography. Data integration and reduction were conducted with SAINT. An empirical absorption correction was applied to the collected reflections with SADABS using XPREP. Structures were determined by direct method using SHELXTL and refined on F^2 by a full-matrix least-squares technique using the SHELXL-97 program package. The lattice parameters and structural data are listed somewhere else in this Supporting Information.

1,3-dimethyl-2-phenyl-2,3-dihydro-1H-benzo[d]imidazole:



MB1-H

In 50 ml Schlenk RB, N^1,N^2-dimethylbenzene-1,2-diamine (0.300 g, 2.202 mmol) was dissolved in 5 ml of dry dichloromethane with activated molecular sieves 3 Å. The solution of benzaldehyde (0.233 g, 2.202 mmol) in dry dichloromethane (5 ml) was slowly added to pre-stirred dichloromethane solution at 0 °C under nitrogen atmosphere. The resulting mixture was stirred at room temperature for 24 h and filtered through cannula under nitrogen atmosphere. The yellow filtrate was concentrated and crystallized in dichloromethane solution. The resulting white crystals was filtered off and dried under high vacuum. (0.050 g, 10%).

^{1}H NMR (400 MHz, $CDCl_3$):

δ_H = 7.58 (m, 2H, Ar), 7.41 (m, 3H, Ar), 6.72 (dd, 2H, Ar), 6.43 (dd, 2H, Ar), 4.88 (s, 1H, CH), 2.57(s 6H, CH_3).

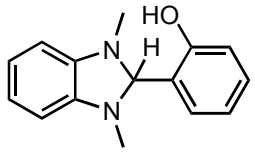
^{13}C NMR (100 MHz, $CDCl_3$):

δ_C = 142.23, 139.20, 129.46, 128.99, 128.60, 119.41, 105.85, 94.18, 33.30.

High Resolution ESI-MS:

m/z for $C_{15}H_{15}N_2$ = 223.1231 (calcd. 223.1235) = $[MB1]^+$.

2-(1,3-dimethyl-2,3-dihydro-1H-benzo[d]imidazol-2-yl)phenol:



MB2-H

In 50 ml Schlenk RB, N^1,N^2-dimethylbenzene-1,2-diamine (0.238 g, 1.747mmol) was dissolved in 5 ml of dry dichloromethane with activated molecular sieves 3 Å. The solution of salicylaldehyde (0.213 g, 1.747mmol) in dry dichloromethane (5 ml) was slowly added to pre-stirred dichloromethane solution at 0 °C under nitrogen atmosphere. The resulting mixture was stirred at room temperature for 20 h and filtered through cannula under nitrogen atmosphere. The yellow filtrate was concentrated and added dry ethanol (5ml). The resulting pale yellow powder was filtered off and dried under high vacuum. (0.060 g, 14%).

^{1}H NMR (400 MHz, DMSO-d6):

δ_H = 9.59 (s, 1H, OH), 7.48 (dd, 1H, Ar), 7.16 (dt, 1H, Ar), 6.85-6.56 (m, 4H, Ar), 6.40 (dd, 2H, Ar), 5.32 (s, 1H, CH), 2.46 (s, 6H, CH_3).

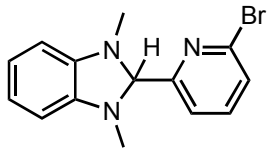
^{13}C NMR (100 MHz, DMSO-d6):

δ_C = 157.97, 143.15, 130.60, 130.11, 124.62, 120.18, 119.97, 116.50, 106.86, 100.44, 34.20.

High Resolution ESI-MS:

m/z for $C_{15}H_{15}N_2O$ = 239.1186 (calcd. 239.1184) = $[MB2]^+$.

2-(6-bromopyridin-2-yl)-1,3-dimethyl-2,3-dihydro-1H-benzo[d]imidazole:



MB3-H

In a oven dried 50 mL Schlenk flask added 2 g of activated 4 Å molecular sieves and 10 mL toluene. It was stirred for 30 minutes at room temperature in N_2 atmosphere, added N^1,N^2-dimethylenzene-1,2-diamine (0.108 g, 0.73 mmol) and the brown solution was heated at 100 ^oC for 2 hours in N_2 atmosphere. It was cooled to room temperature and 6-bromopyridine-2-carboxaldehyde (0.136 g) was added as solid, immediately colour of the solution turned from brown to yellow. The yellow solution was heated at 100 ^oC for 12 hours and refluxed at 130 ^oC for 3 hours in N_2 atmosphere. The yellow solution cannulated to 50 mL round bottom flask and the solvent was removed under reduced pressure gave orange yellow oil. It was dried under high vacuum for 1 hour gave orange yellow solid, which was dissolved in diethyl ether and left in -35 ^oC forms orange crystals suitable for single crystal X-ray diffraction. (0.208 g, 94%).

^{1}H NMR (400MHz, $CDCl_3$):

δ_H = 7.83 (d, 1H, Py), 7.66 (t, 1H, Py), 7.52 (d, 1H, Py), 6.74 (dd, 2H, Ph), 6.45 (dd, 2H, Ph), 5.13 (s, 1H), 2.67 (s, 6H).

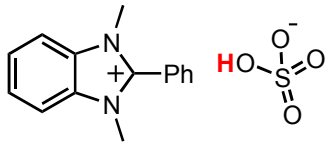
^{13}C NMR (100MHz, $CDCl_3$):

δ_C = 161.68, 141.98, 141.03, 139.60, 128.26, 121.54, 119.69, 106.10, 93.50, 33.96.

High Resolution ESI-MS:

m/z for $C_{14}H_{13}BrN_3$ = 302.0284 (calcd. 302.0293) = $[MB3]^+$.

Reaction of MB1-H with SO_2:



Yellow crystalline powder of HL (0.028 g, 0.124 mmol) was dissolved in dry acetonitrile (5 ml) and purged SO_2 at room temperature for 20 min. While purging SO_2, the color of the solution turned to bright red. The resulting solution was stored at -30 °C for one day and evaporated the solvent to get brick red precipitate. Crystals were grown in mixture of methanol and acetonitrile at -30 °C (0.020 g, 51%).

^{1}H NMR (400 MHz, DMSO-d6):

δ_H = 8.15 (dt, 2H, Ar), 7.91 (m, 2H, Ar), 7.83 (m, 5H, Ar), 3.89 (s, 6H, CH_3).

^{13}C NMR (100 MHz, DMSO-d6):

δ_C = 151.32, 133.92, 132.70, 131.73, 130.44, 127.62, 121.99, 114.39, 33.79.

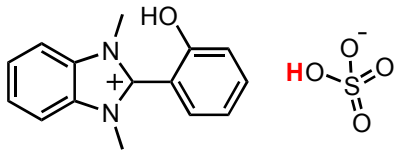
High Resolution ESI-MS (cation mode):

m/z for $C_{15}H_{15}N_2$ = 223.1236 (calcd. 223.1235) = $MB1^+$ cation.

High Resolution ESI-MS (anion mode):

m/z for HSO_4 = 96.9582 (calcd. 96.9596) = bisulfate anion

Reaction of MB2-H with SO_2:



Yellow crystalline powder of HL (0.060 g, 0.249 mmol) was dissolved in dry acetonitrile (4 ml) and purged SO_2 at room temperature for 20 min. While purging SO_2, the color of the solution turned to brown. The resulting solution was stored at -30 °C for one day and crystals were grown in one week at room temperature (0.076 g, 91%).

^{1}H NMR (400 MHz, DMSO-d6):

δ = 11.04 (s, 1H, OH), 8.12 (dd, 2H, Ar), 7.76 (dd, 2H, Ar), 7.67 (m, 2H, Ar), 7.22 (m, 2H, Ar), 3.87 (s, 6H, CH_3).

^{13}C NMR (100 MHz, DMSO-d6):

δ157.58, 149.98, 135.96, 132.72, 132.63, 127.56, 120.87, 117.84, 114.34, 108.47, 33.54.

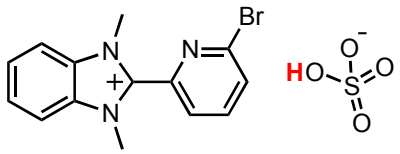
High Resolution ESI-MS (cation mode):

m/z = 239.1167 (calcd. 239.1184) = $MB2^+$ cation.

High Resolution ESI-MS (anion mode):

m/z for HSO_4 = 96.9572 (calcd. 96.9596) = bisulfate anion

Reaction of MB-3 with SO_2:



Yellow powder of HL (37 mg, 0.121 mmol) was dissolved in dry acetonitrile (4 ml) and purged SO_2 at room temperature for 20 min. While purging SO_2, the color of the solution turned to brown. The resulting solution was stored at -30 °C for one day and crystals were formed immediately at -30 °C (0.041 mg, 85%).

^{1}H NMR (400 MHz, DMSO d6):

δ_H = 8.19 (d, 2H, Ph), 8.13-8.10 (m, 3H, py), 7.78 (dd, 2H, Ph), 4.06 (s, 6H, CH_3).

^{13}C NMR (100 MHz, DMSO-d6):

δ_C = 146.80, 142.93, 142.18, 142.14, 132.98, 132.62, 128.94, 128.25, 114.69, 34.18.

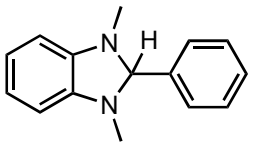
High Resolution ESI-MS (cation mode):

m/z = 302.0290 (calcd. 302.0293) = $MB3^+$ cation.

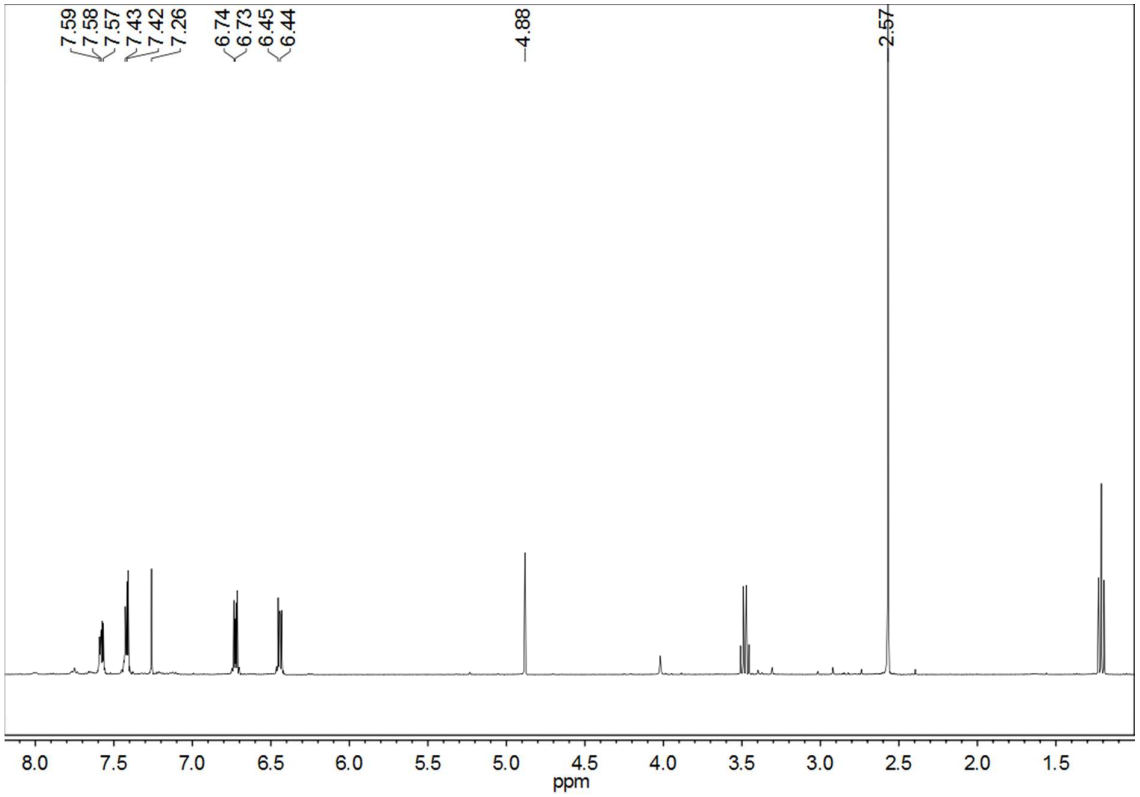
High Resolution ESI-MS (anion mode):

m/z for HSO_4 = 96.9590 (calcd. 96.9596) = bisulfate anion

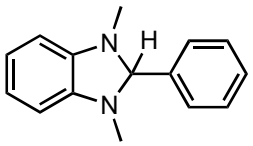
Figure S 1. ^{1}H NMR of MB1-H in $CDCl_3$.



MB1-H



SFigure S 2. ^{13}C NMR of MB1-H in $CDCl_3$.



MB1-H

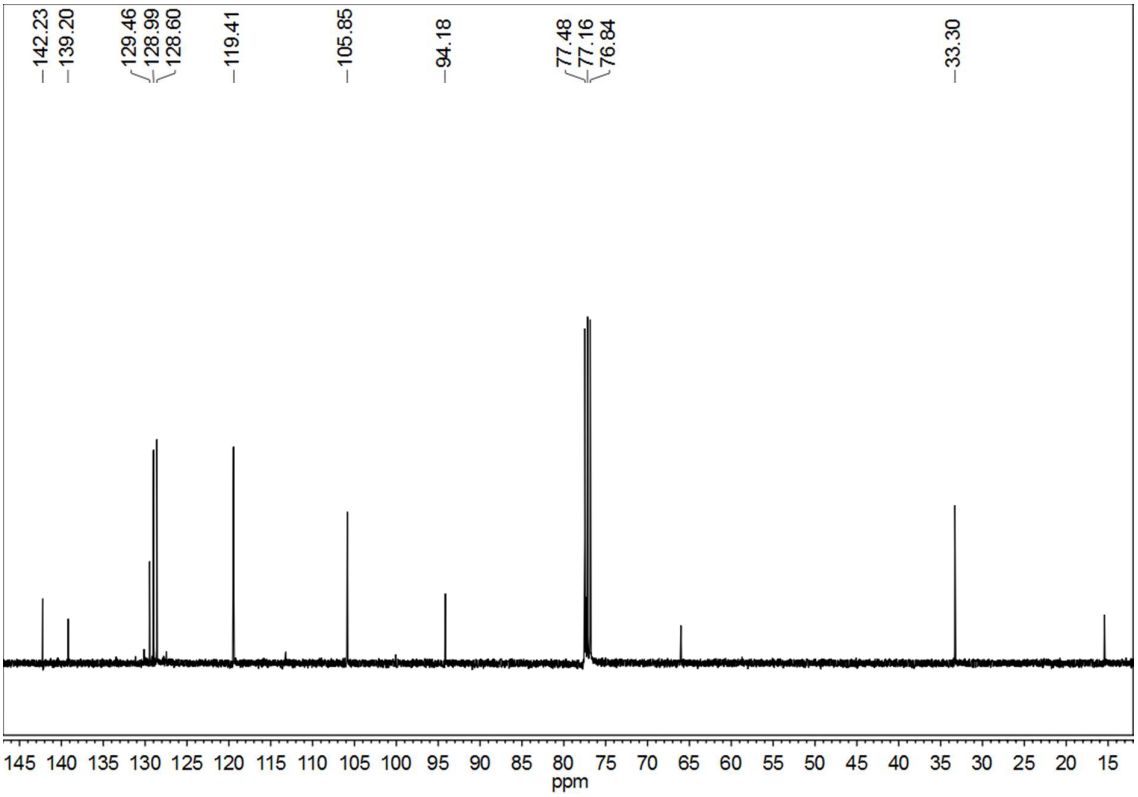
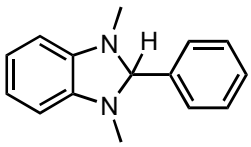


Figure S 3. ESI-MS of MB1-H.

$[M-H]^+ = C_{15}H_{15}N_2 = 223.1231$ (obs); 223.1235 (calcd.).



MB1-H

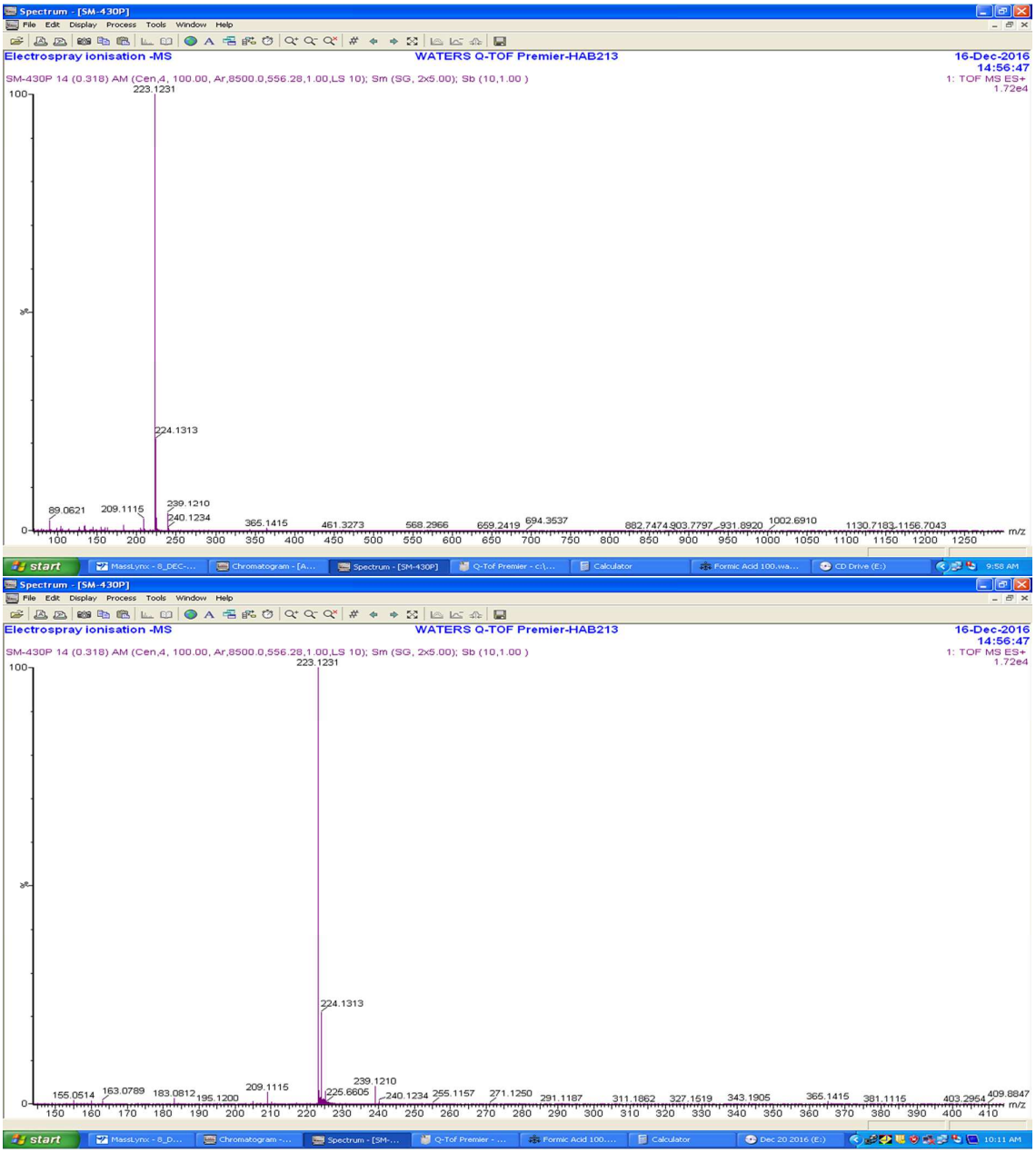
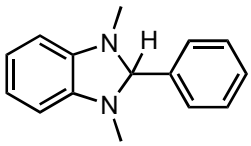


Figure S 4. Isotopic distribution simulated for $MB1^+$ cation.



MB1-H

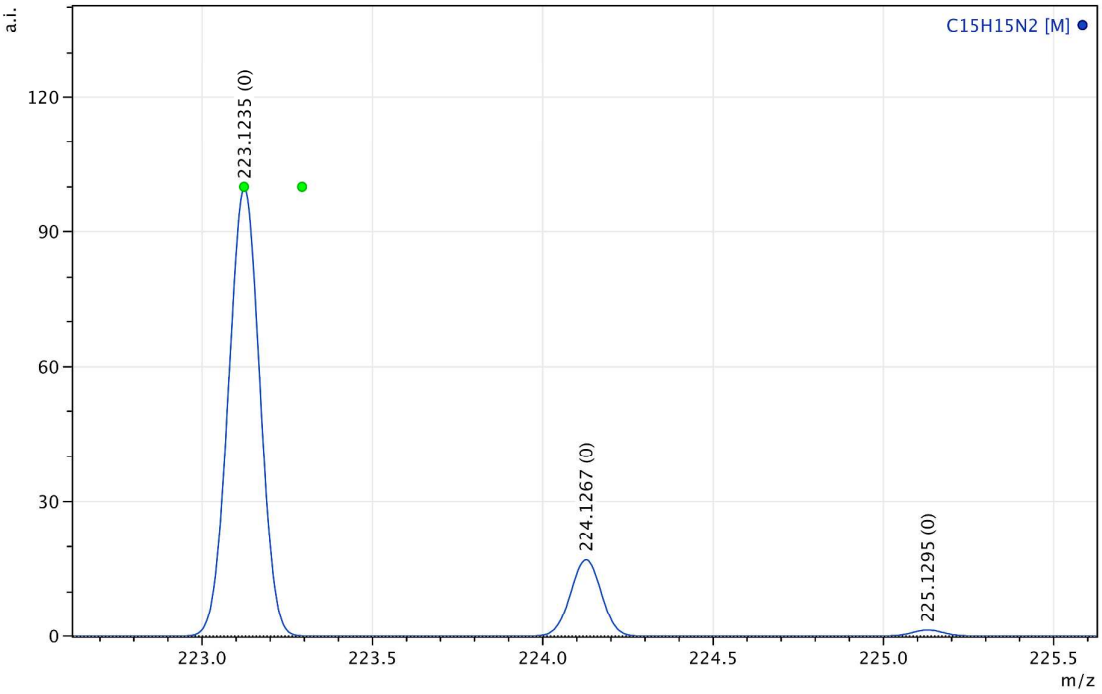


Figure S 5. ^{1}H NMR of [MB1](HSO_4).

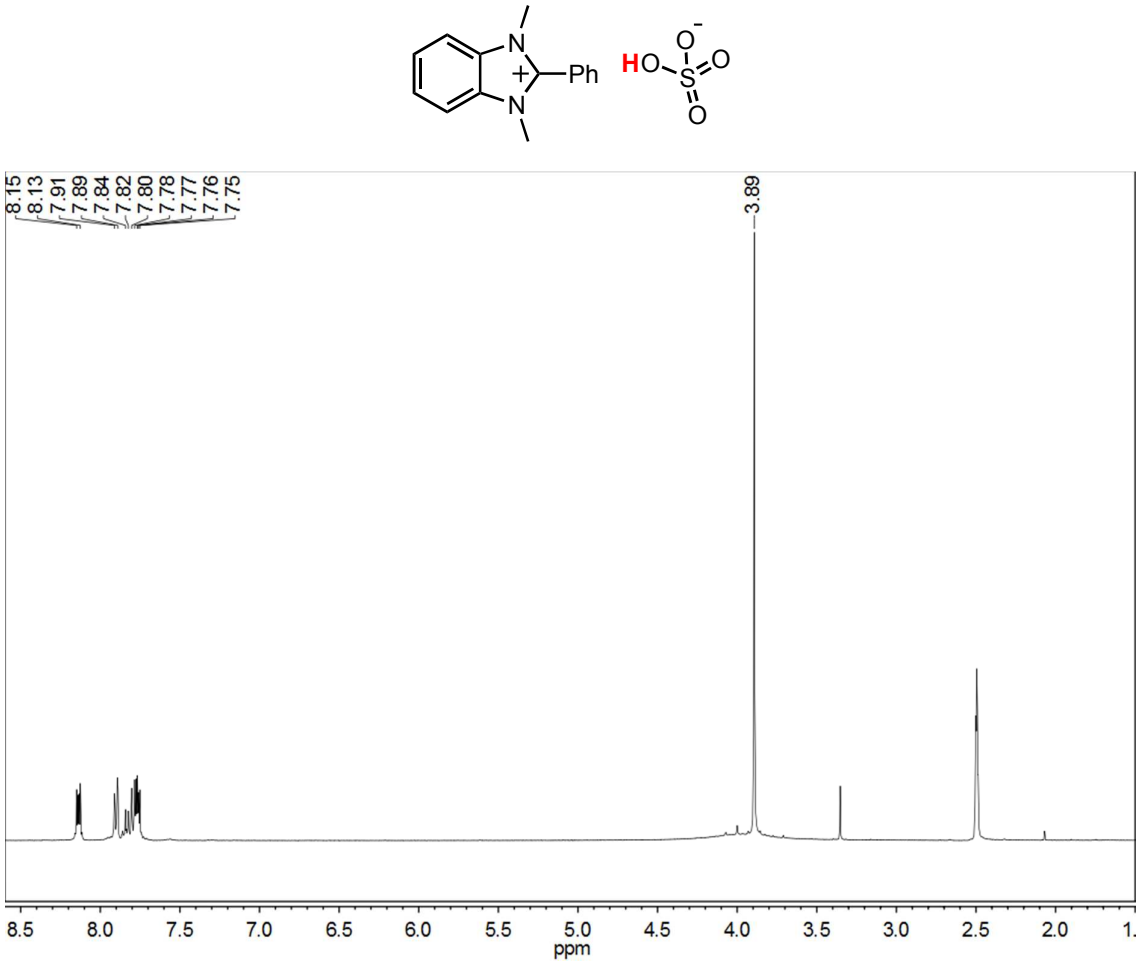


Figure S 6. ^{13}C NMR of [MB1](HSO_4).

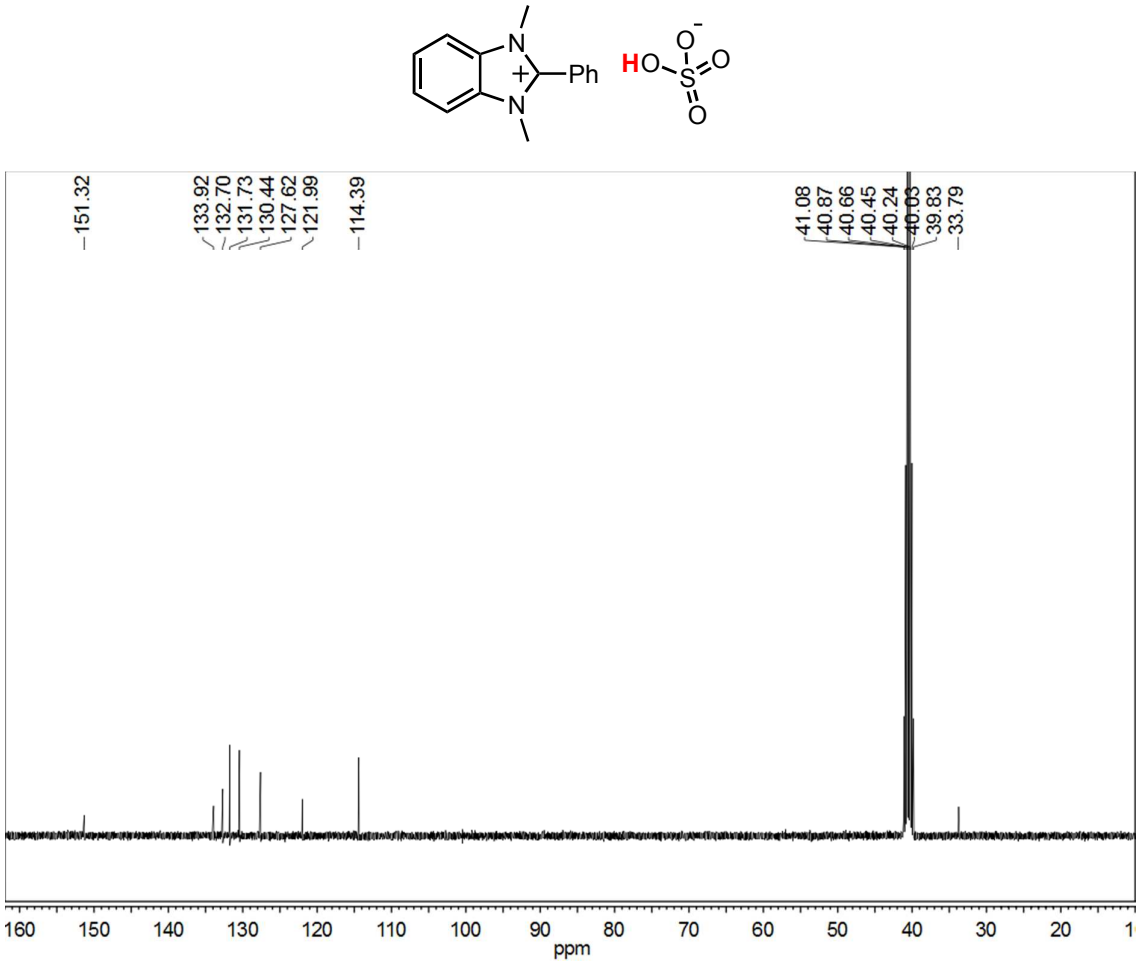
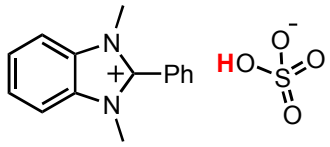
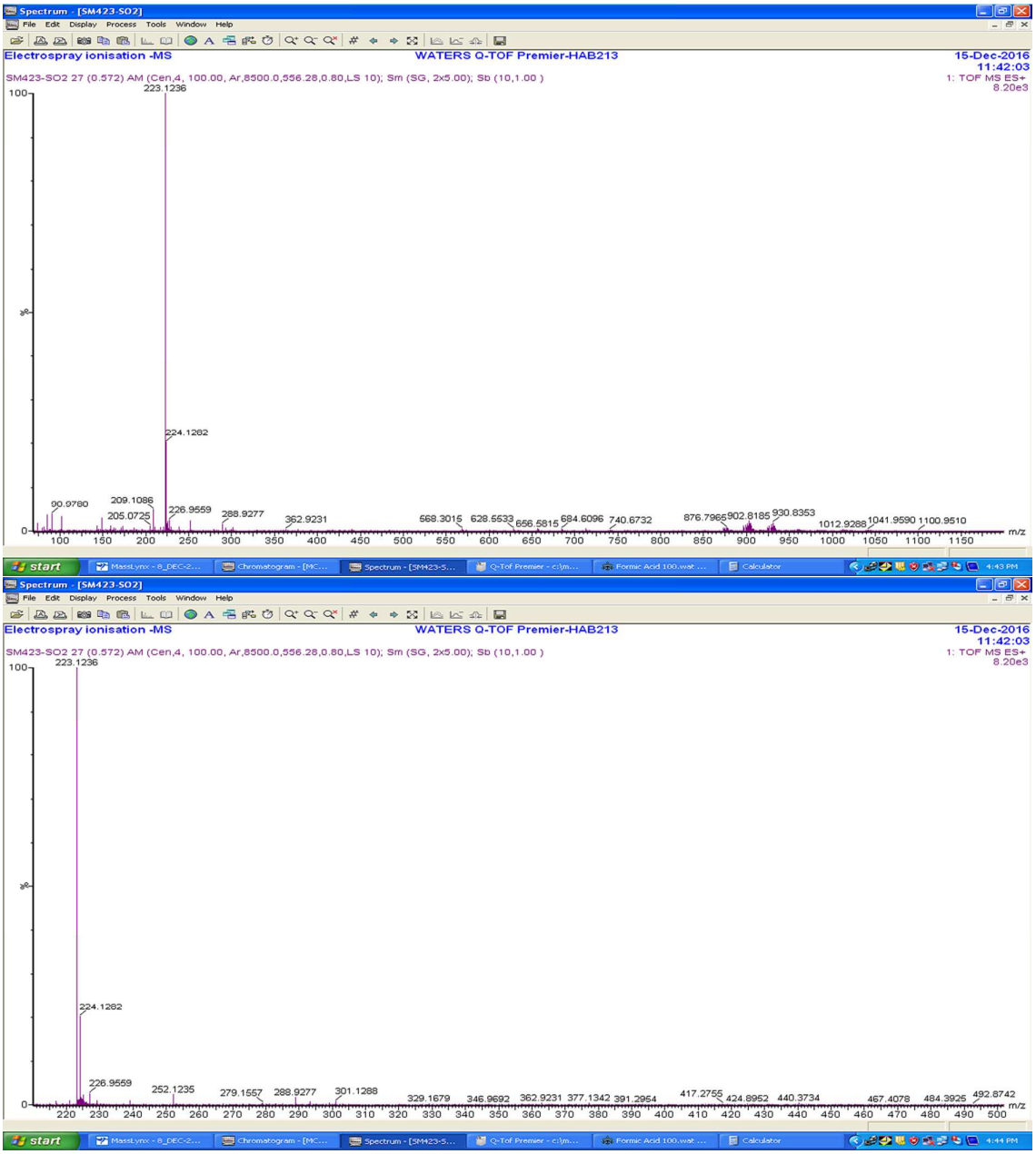


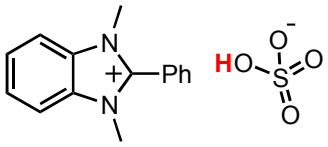
Figure S 7. ESI-MS of [MB1](HSO$_4$) (**cation mode**).





See **Figure S 4** for simulated isotopic distribution.

Figure S 8. ESI-MS of [MB1](HSO$_4$) (**anion mode**).



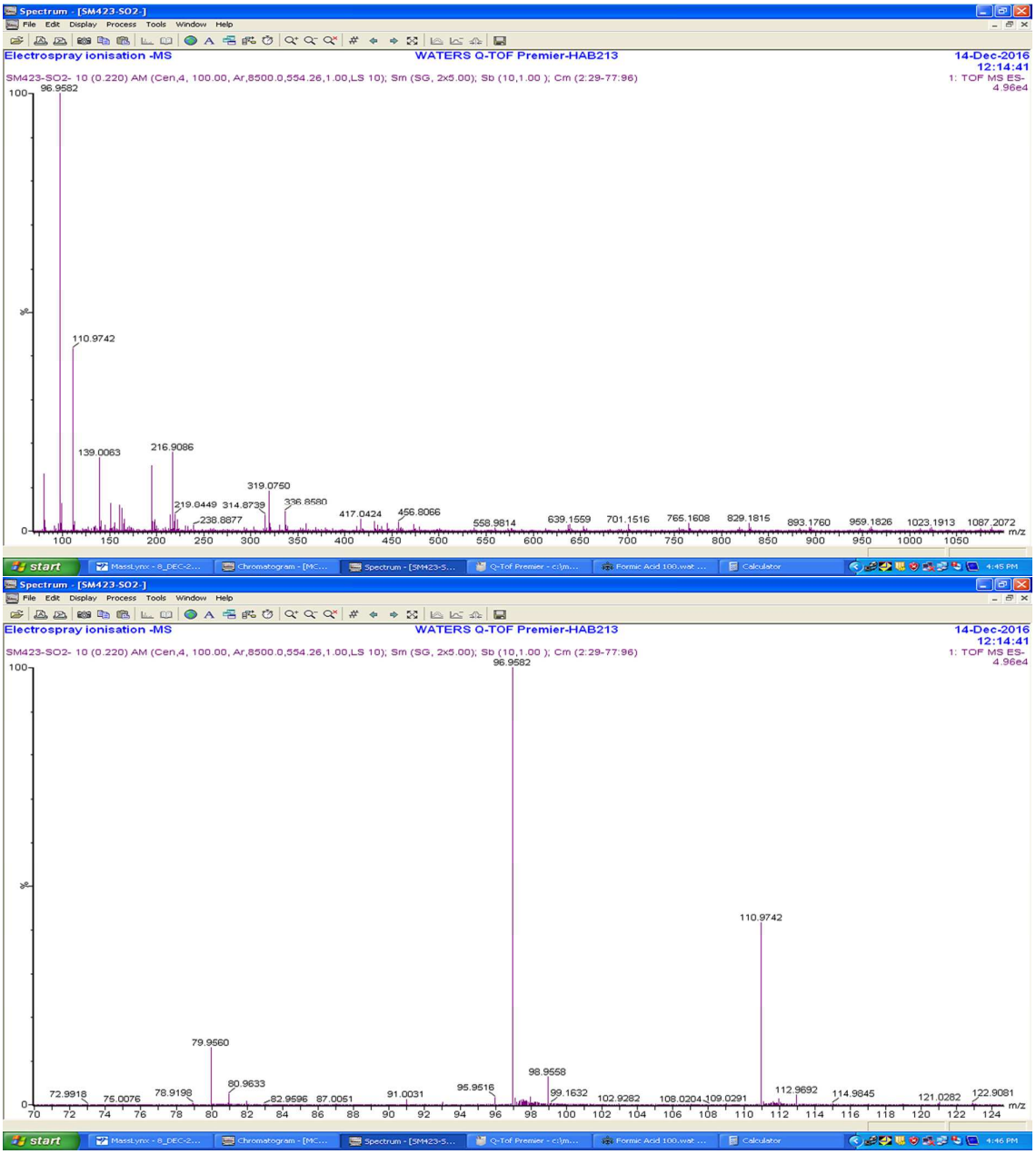
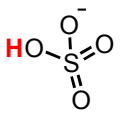


Figure S 9. Isotopic distribution simulated for bisulphate anion.



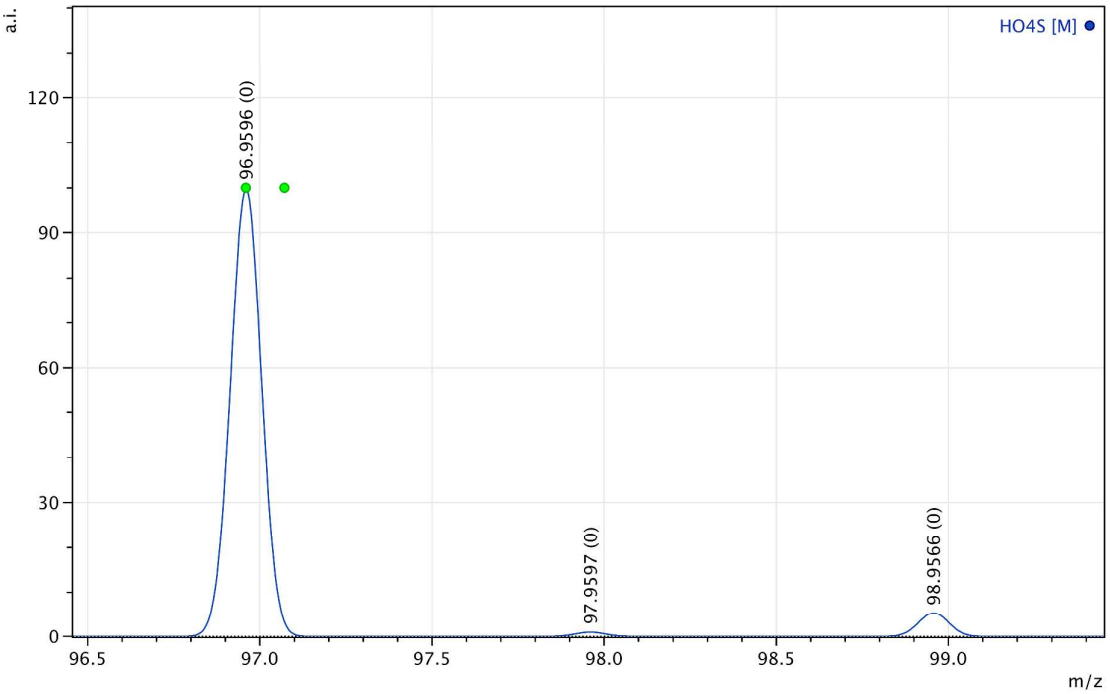
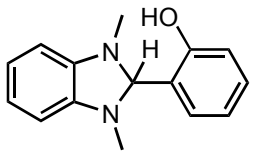


Figure S 10. ^{1}H NMR of MB2-H in DMSO-d6.



MB2-H

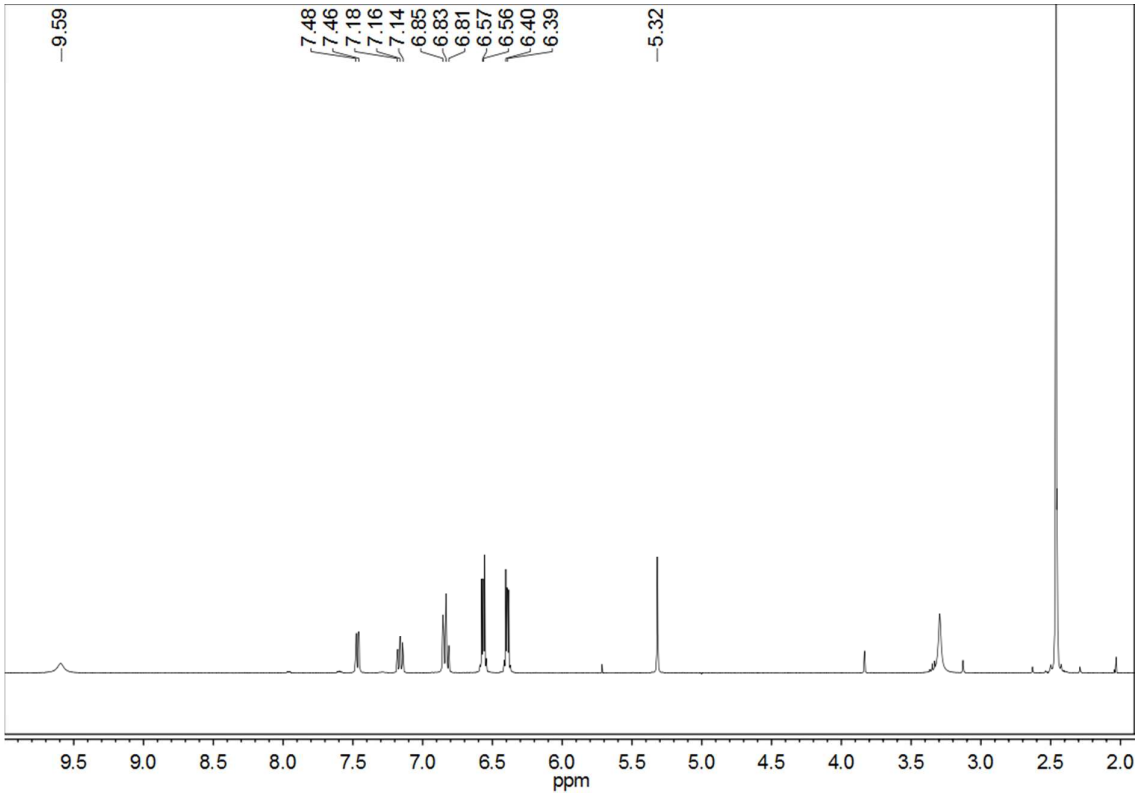
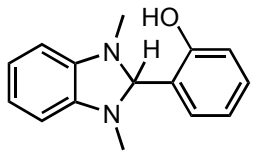


Figure S 11. ^{13}C NMR of MB2-H in DMSO-d6.



MB2-H

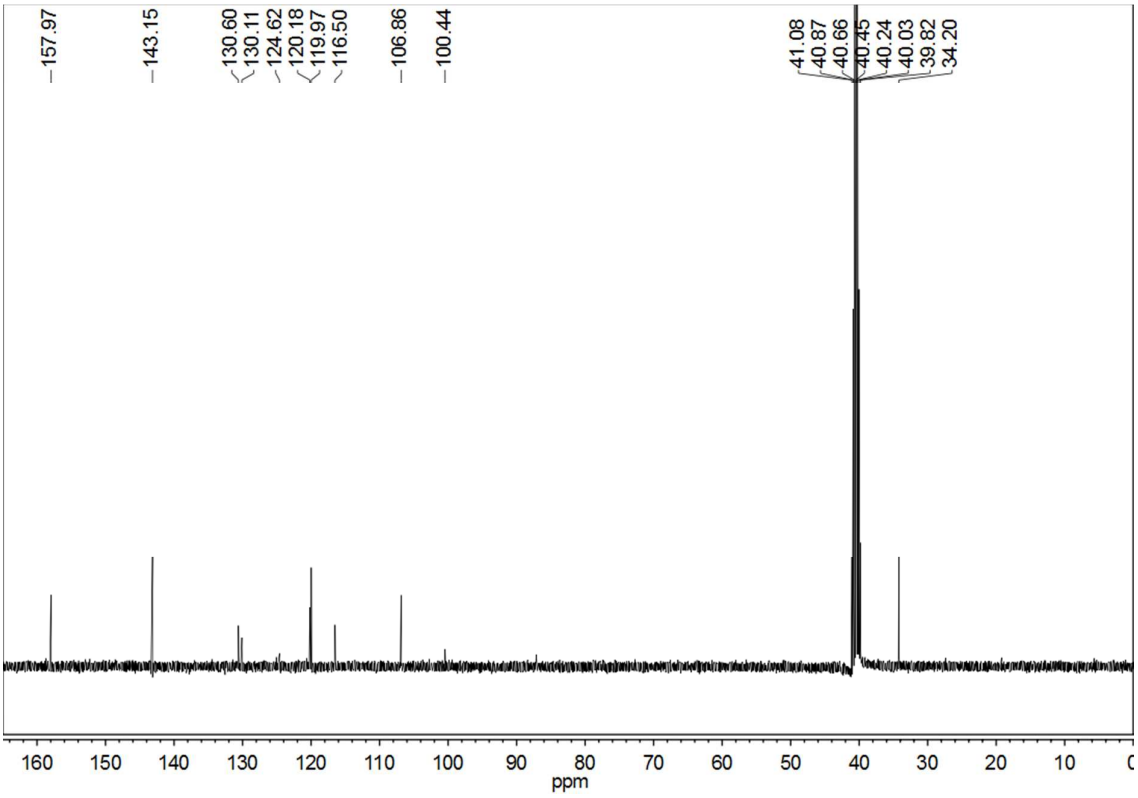
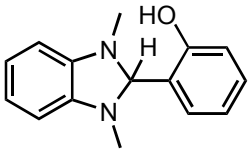


Figure S 12. ESI-MS of MB2-H.

$[M-H]^+$ = $C_{15}H_{15}N_2O$ = 239.1186 (obs); 239.1184 (calcd.).



MB2-H

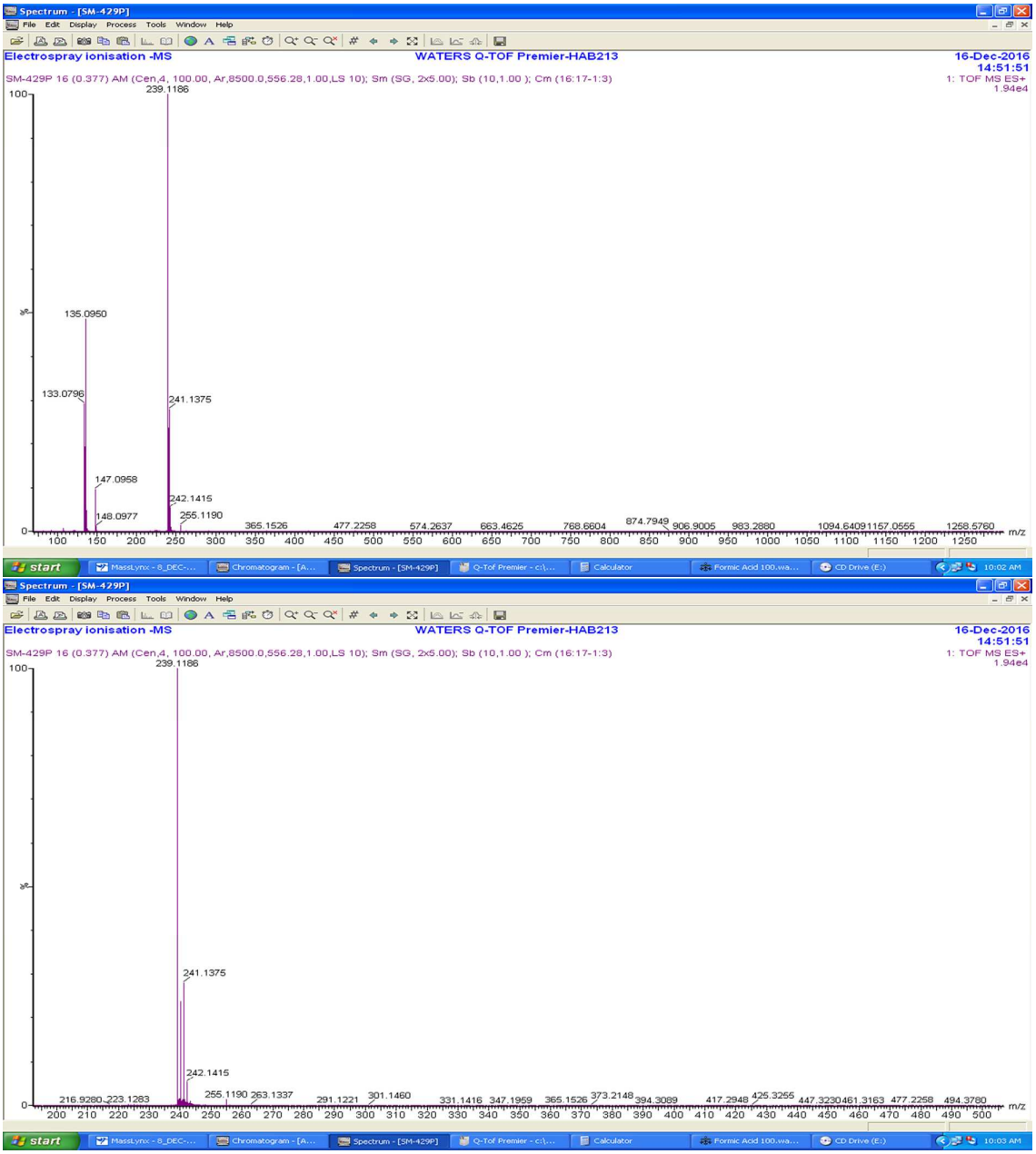
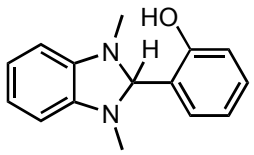


Figure S 13. Isotopic distribution simulated for $MB2^{+}$ cation.



MB2-H

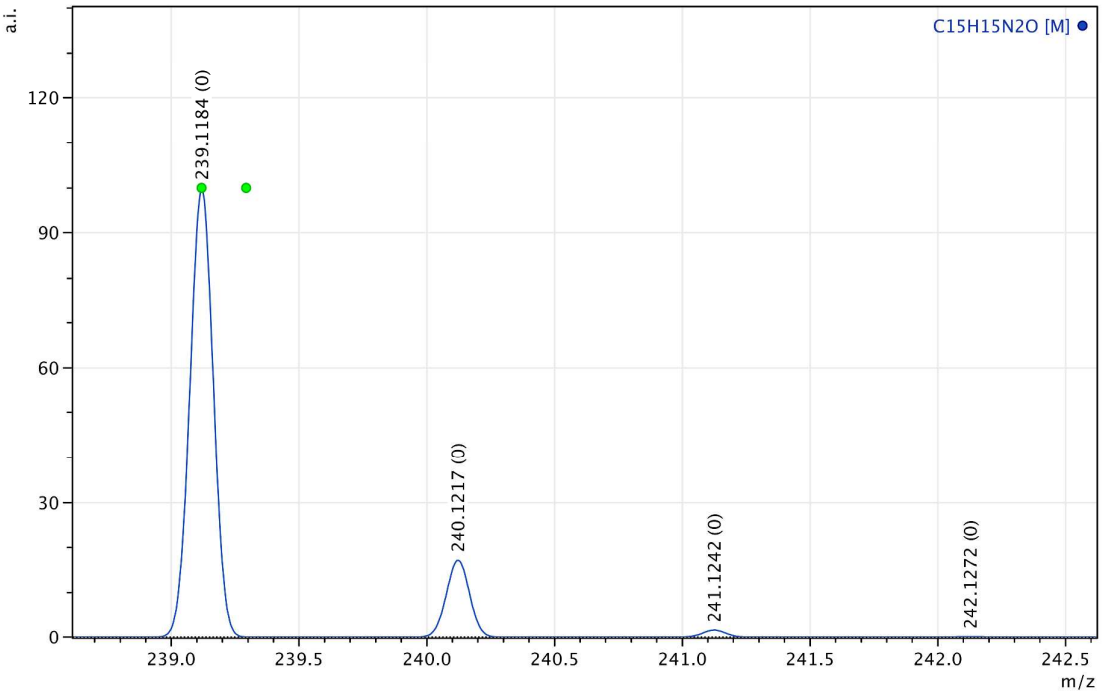


Figure S 14. ^{1}H NMR of [MB2](HSO_4) in DMSO-d6.

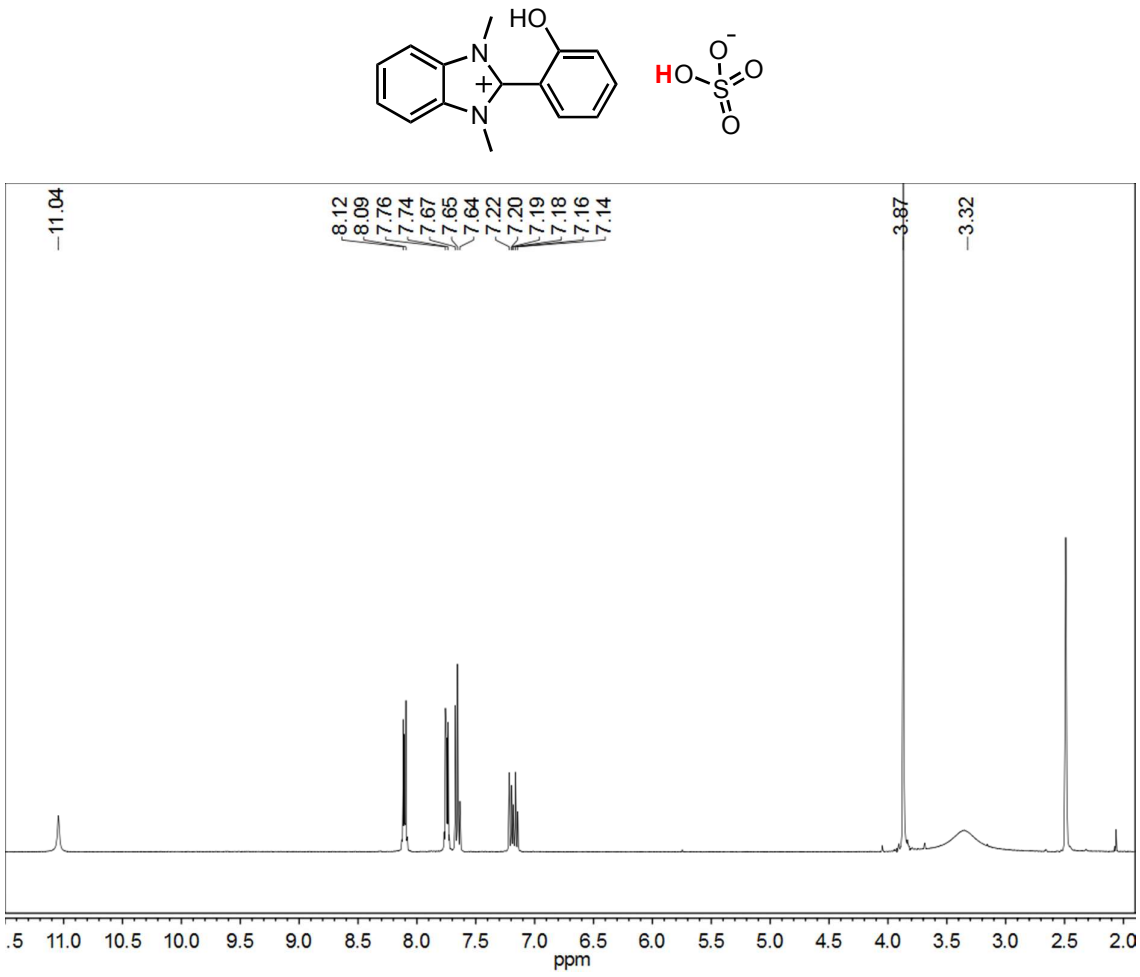


Figure S 15. ^{13}C NMR [MB2](HSO_4) in DMSO-d6.

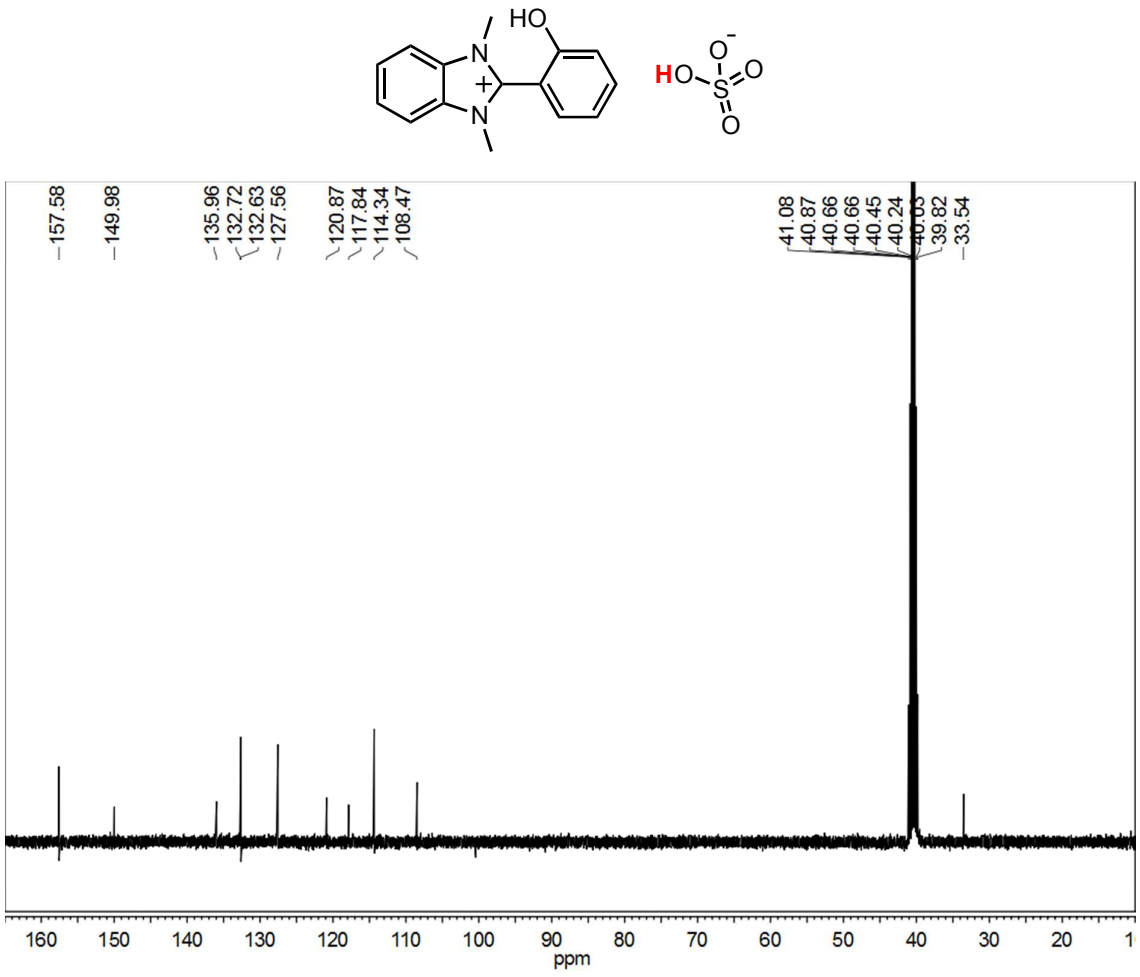
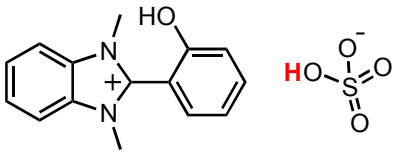
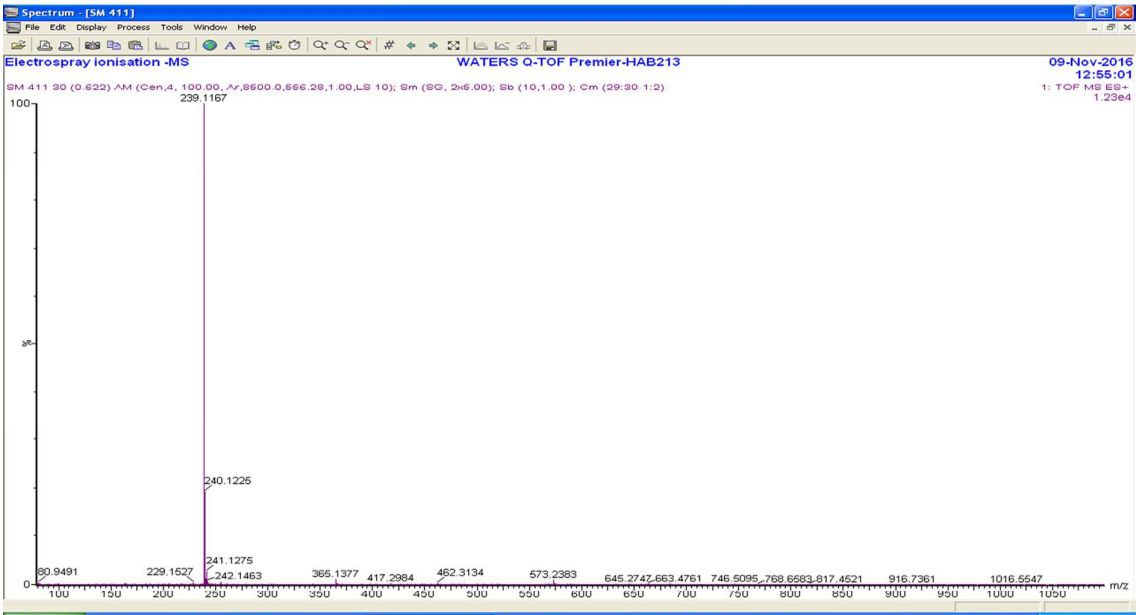
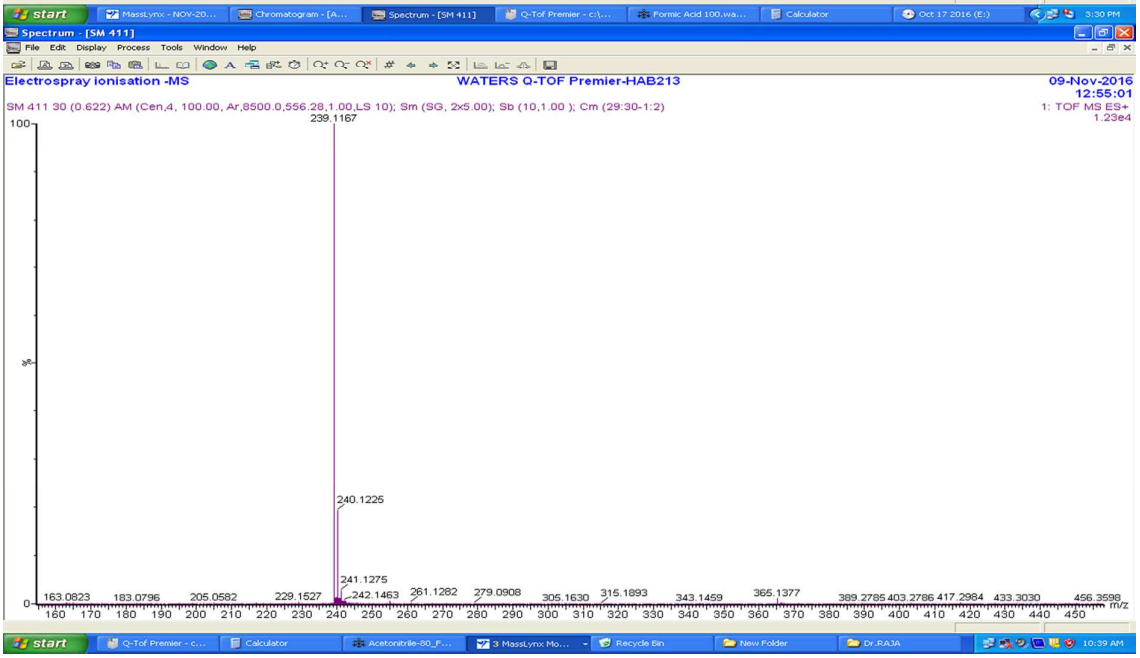


Figure S 16. ESI-MS of [MB2](HSO$_4$) **(cation mode).**

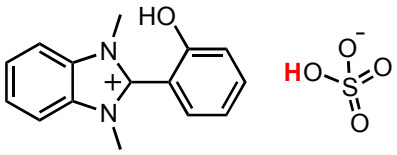


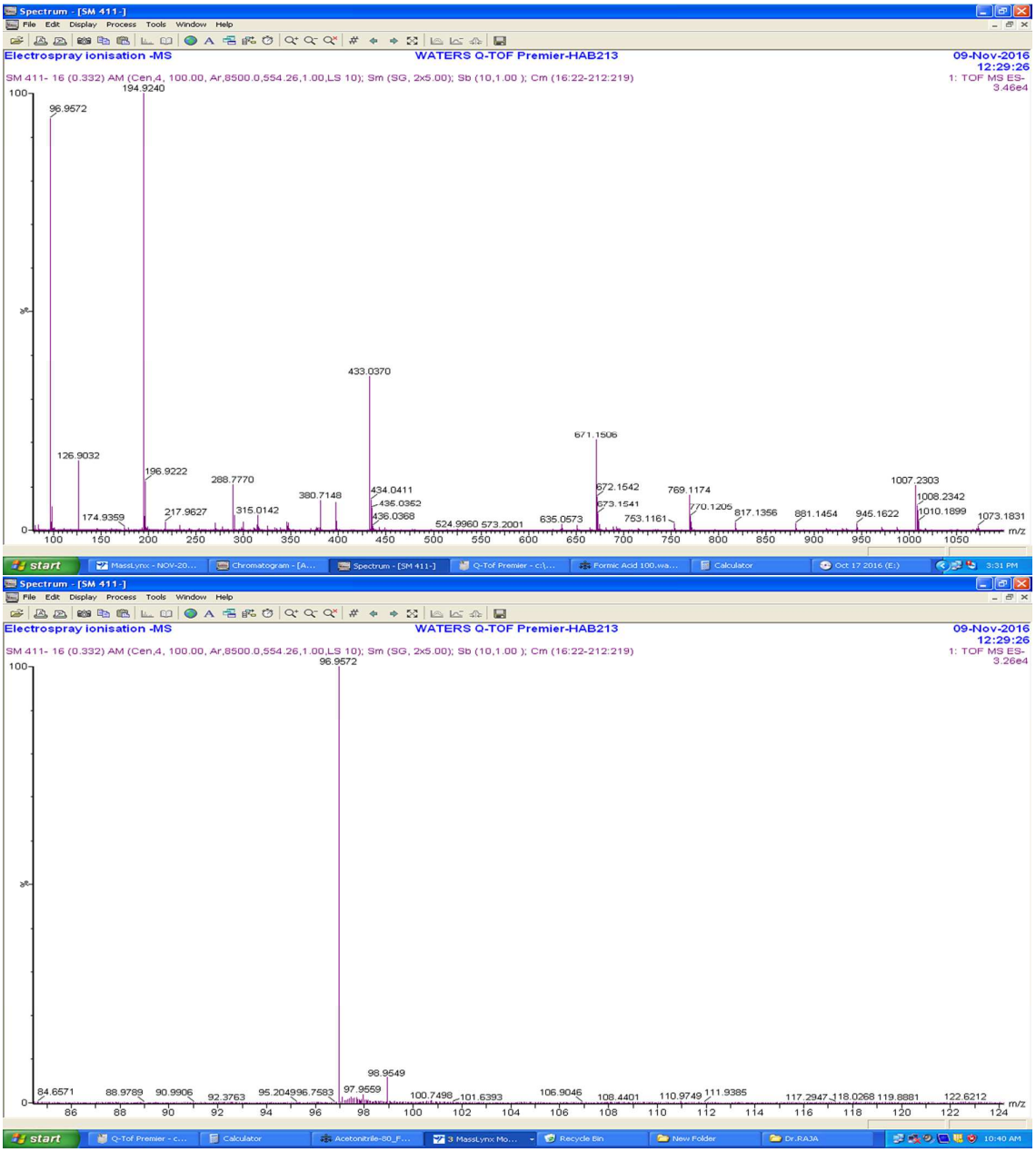




See Figure S 13 for simulated isotopic distribution.

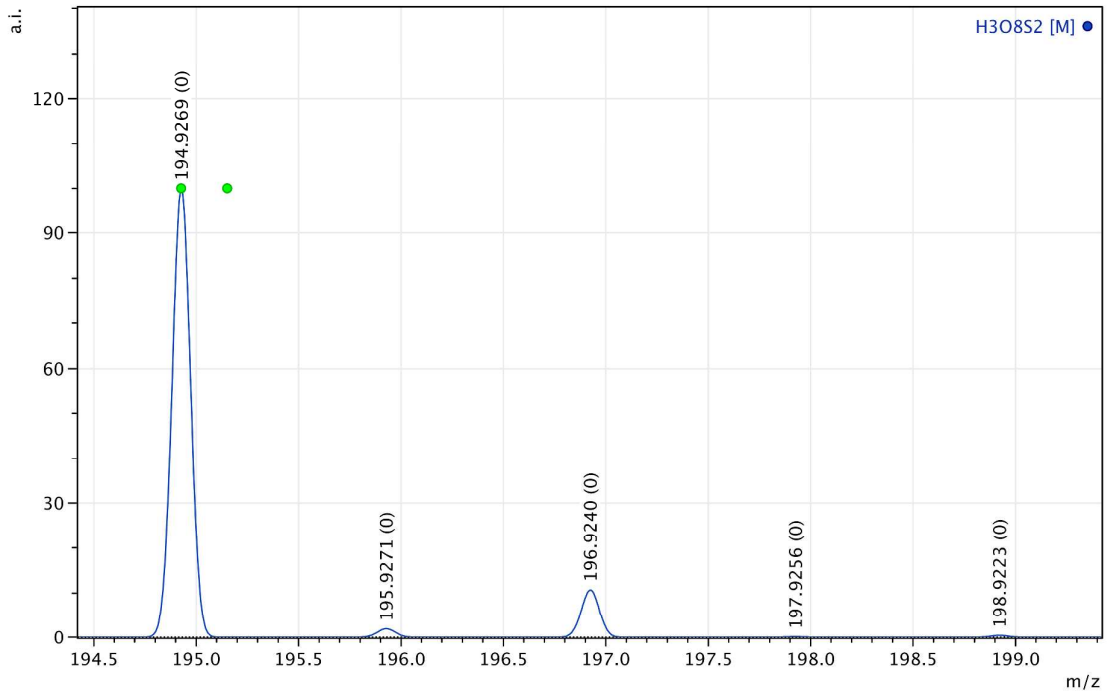
Figure S 17. ESI-MS of [MB2](HSO_4) (**anion mode**).





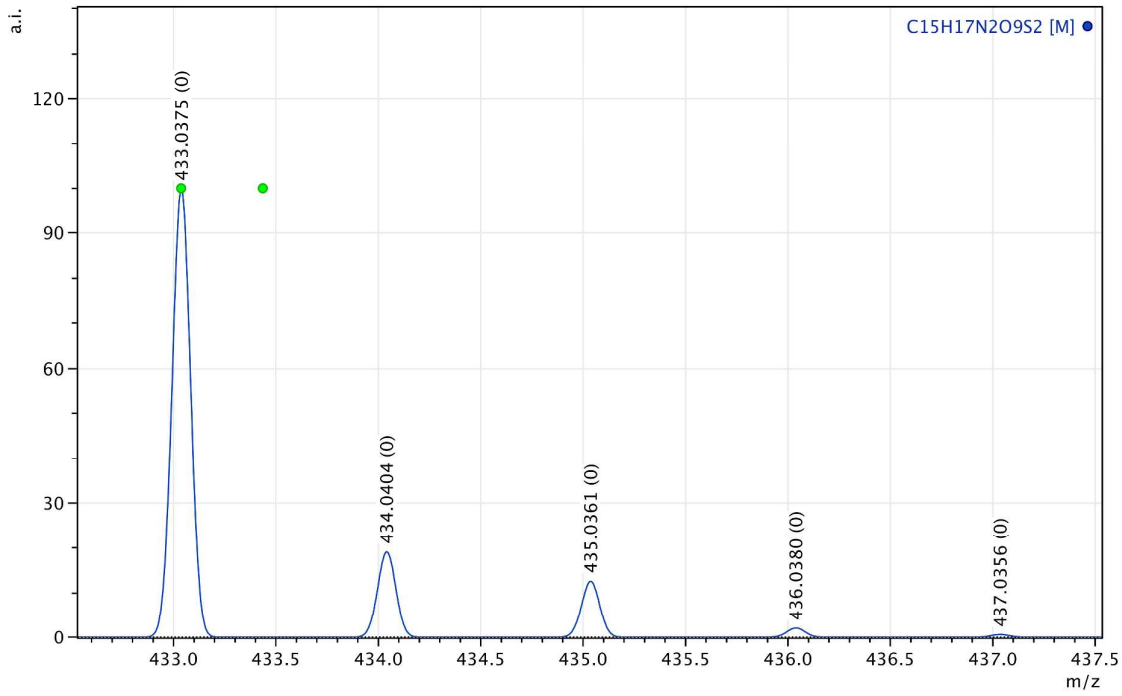
See Figure S 9, Figure S 18, Figure S 19, Figure S 20, Figure S 21, Figure S 22 for simulated isotopic distribution.

Figure S 18. Isotopic distribution simulated for $[HSO_4^{-}\cdots HSO_4^{-} + H^{+}]^{-}$.



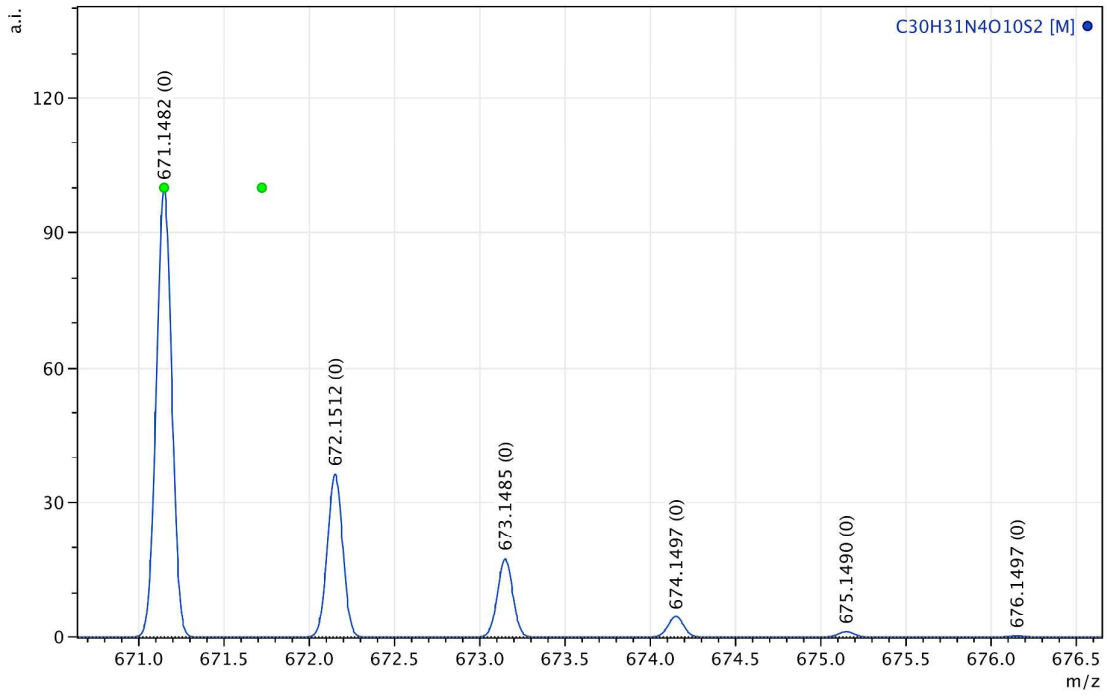
Observed at 194.9240 (calculated *m/z* = 194.9269). See **Figure S 17**.

Figure S 19. Isotopic distribution simulated for $[MB2^{+}\cdots(HSO_4^{-}\cdots HSO_4^{-})]^{-}$.



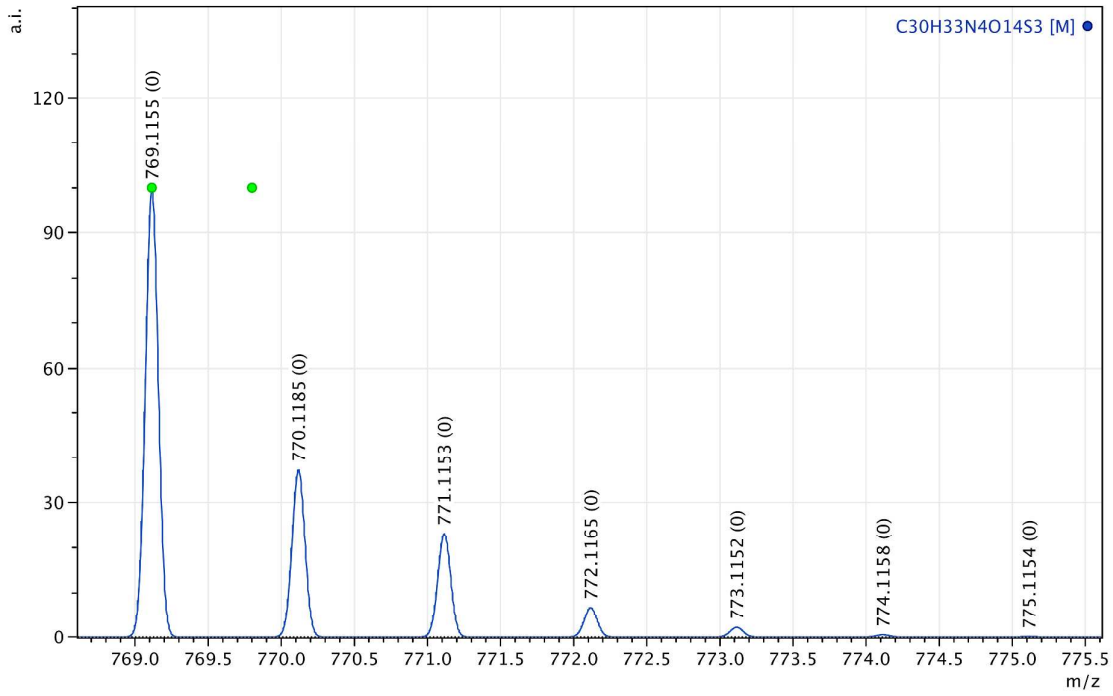
Observed at 433.0370 (calculated *m/z* = 433.075). See **Figure S 17**.

Figure S 20. Isotopic distribution simulated for $[(MB2HSO_4)_2 - H^+]^-$.



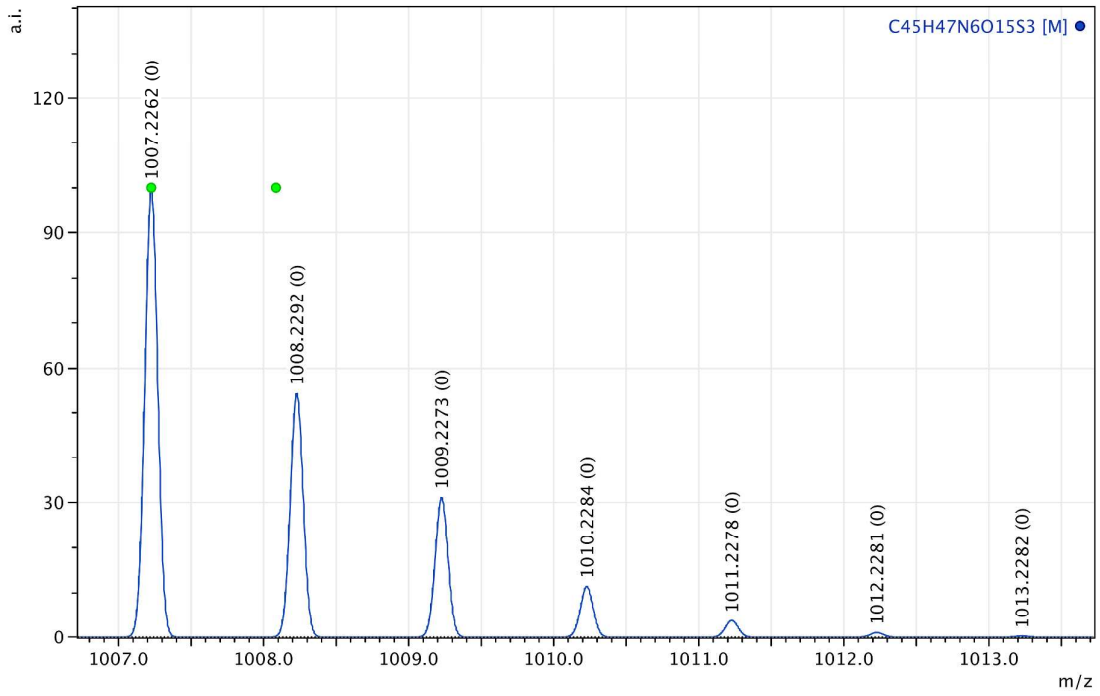
Observed at 671.1506 (calculated *m/z* = 671.1482). **Figure S 17**.

Figure S 21. Isotopic distribution simulated for $[(MB2HSO_4)_2 + HSO_4]^-$.



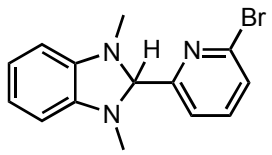
Observed at 769.1174 (calculated *m/z* = 769.1155). **Figure S 17**.

Figure S 22. Isotopic distribution simulated for $[(MB2HSO_4)_3 - H^+]^-$



Observed at 1007.2303 (calculated *m/z* = 1007.2262). See **Figure S 17**.

Figure S 23. ^{1}H NMR of MB3-H in $CDCl_3$.



MB3-H

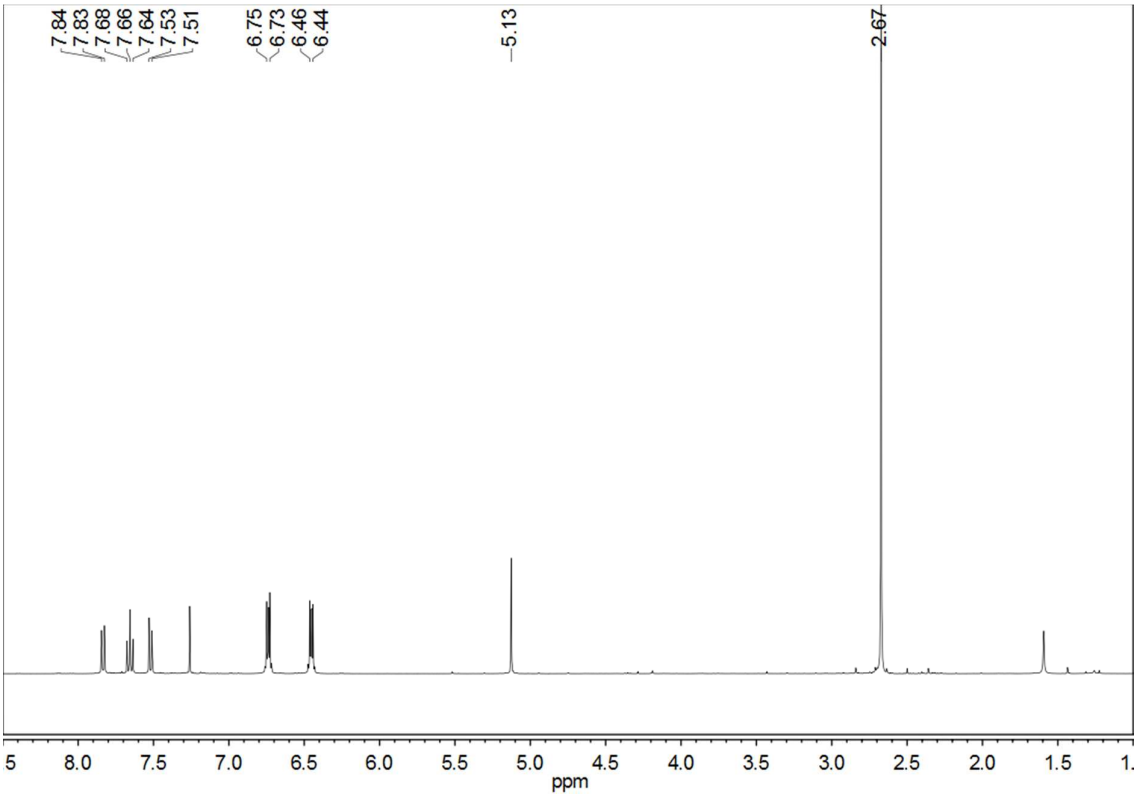
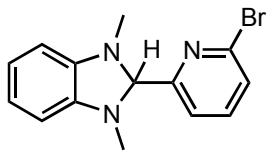


Figure S 24. ^{13}C NMR MB3-H in $CDCl_3$.



MB3-H

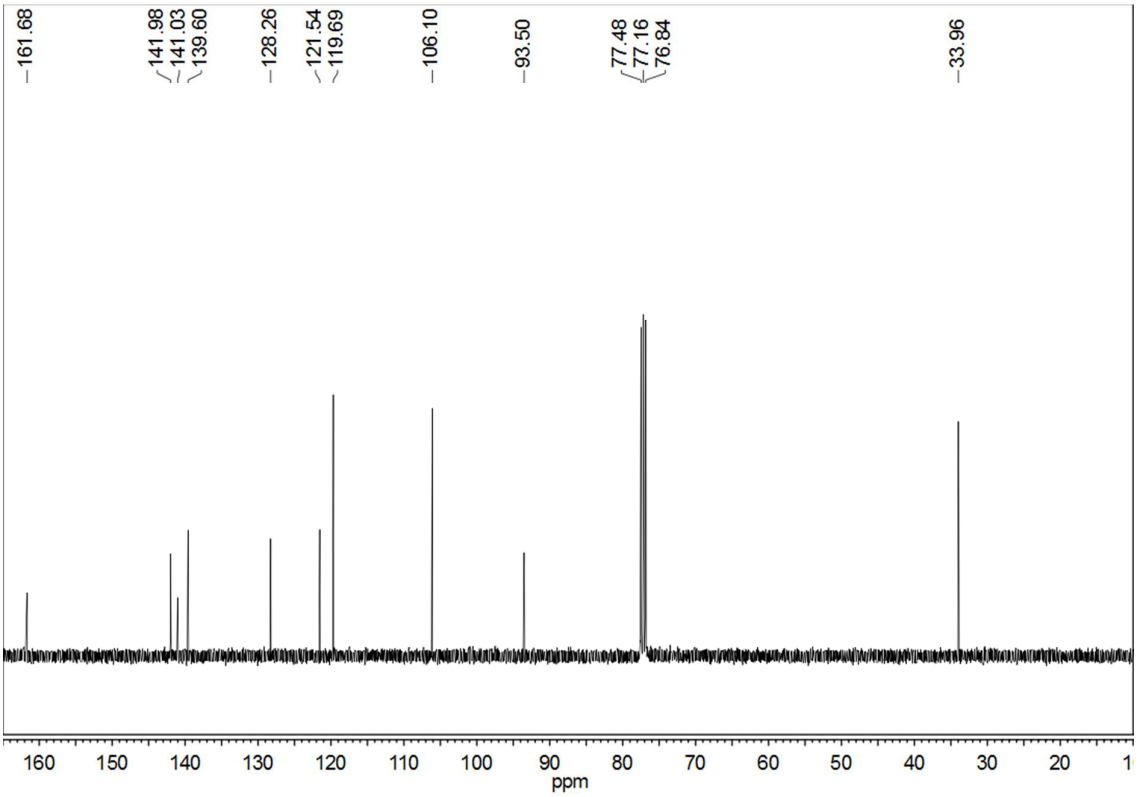
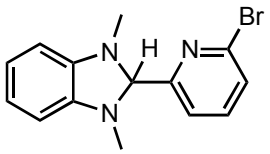


Figure S 25. ESI-MS of MB3-H.

$[M-H]^+ = C_{14}H_{13}BrN_3 = 302.0284$ (obs); 302.0293 (calcd.).



MB3-H

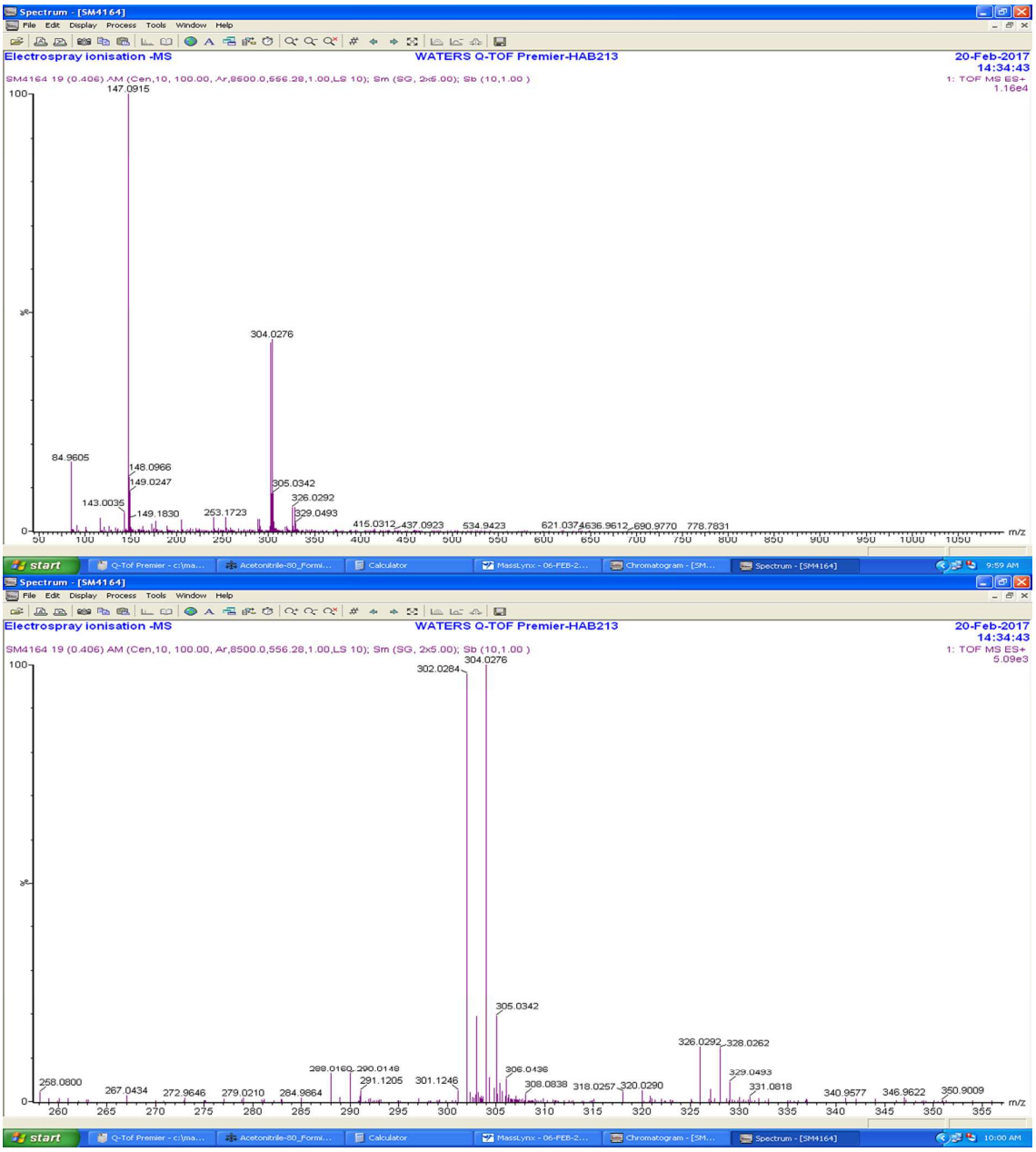


Figure S 26. Isotopic distribution simulated for $MB3^+$ cation.

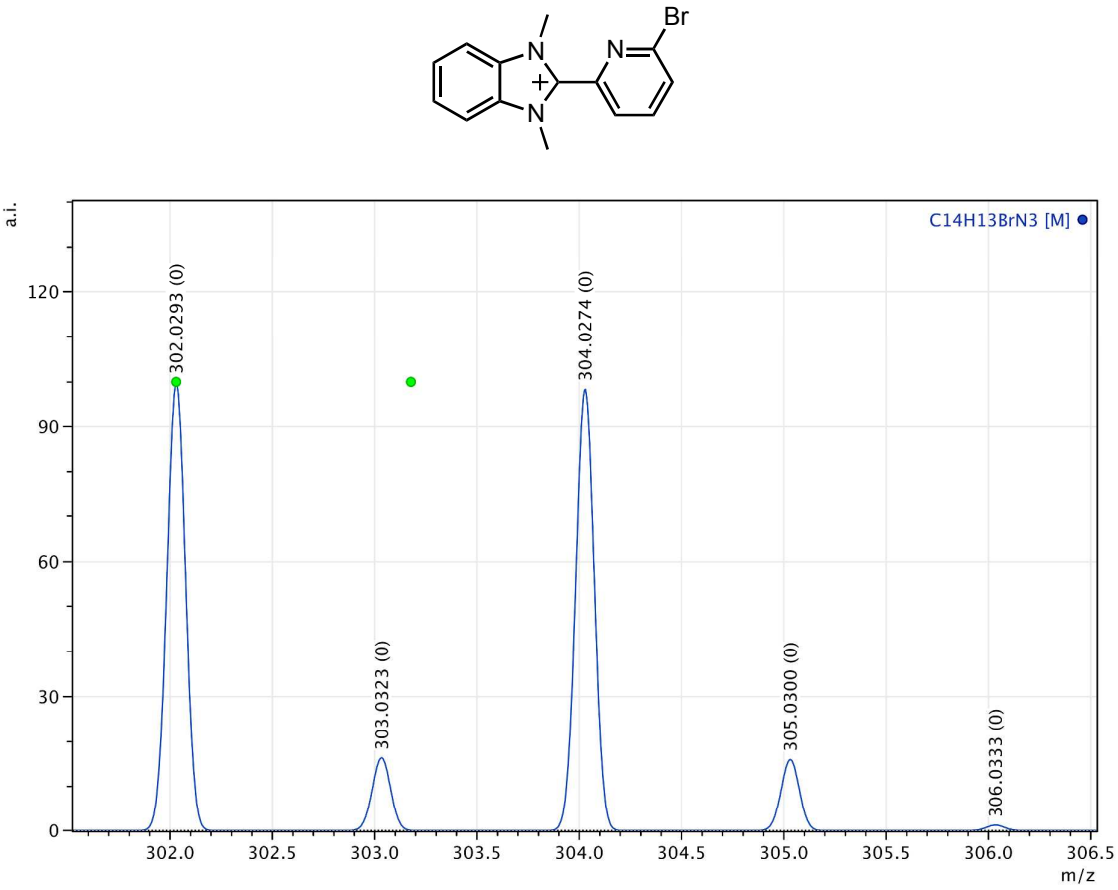


Figure S 27. ^{1}H NMR of [MB3](HSO$_4$) in DMSO-d6.

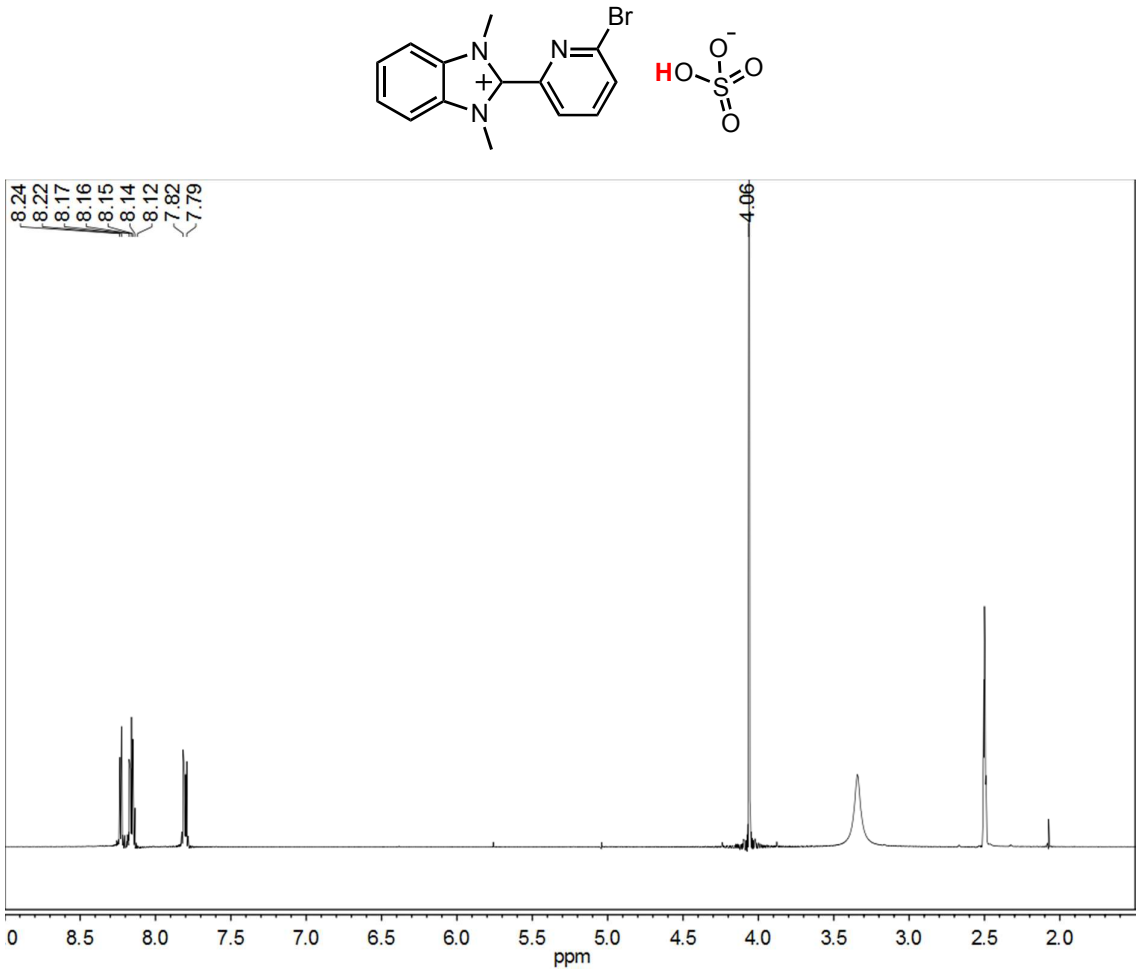


Figure S 28. ^{13}C NMR of [MB3](HSO_4) in DMSO-d6.

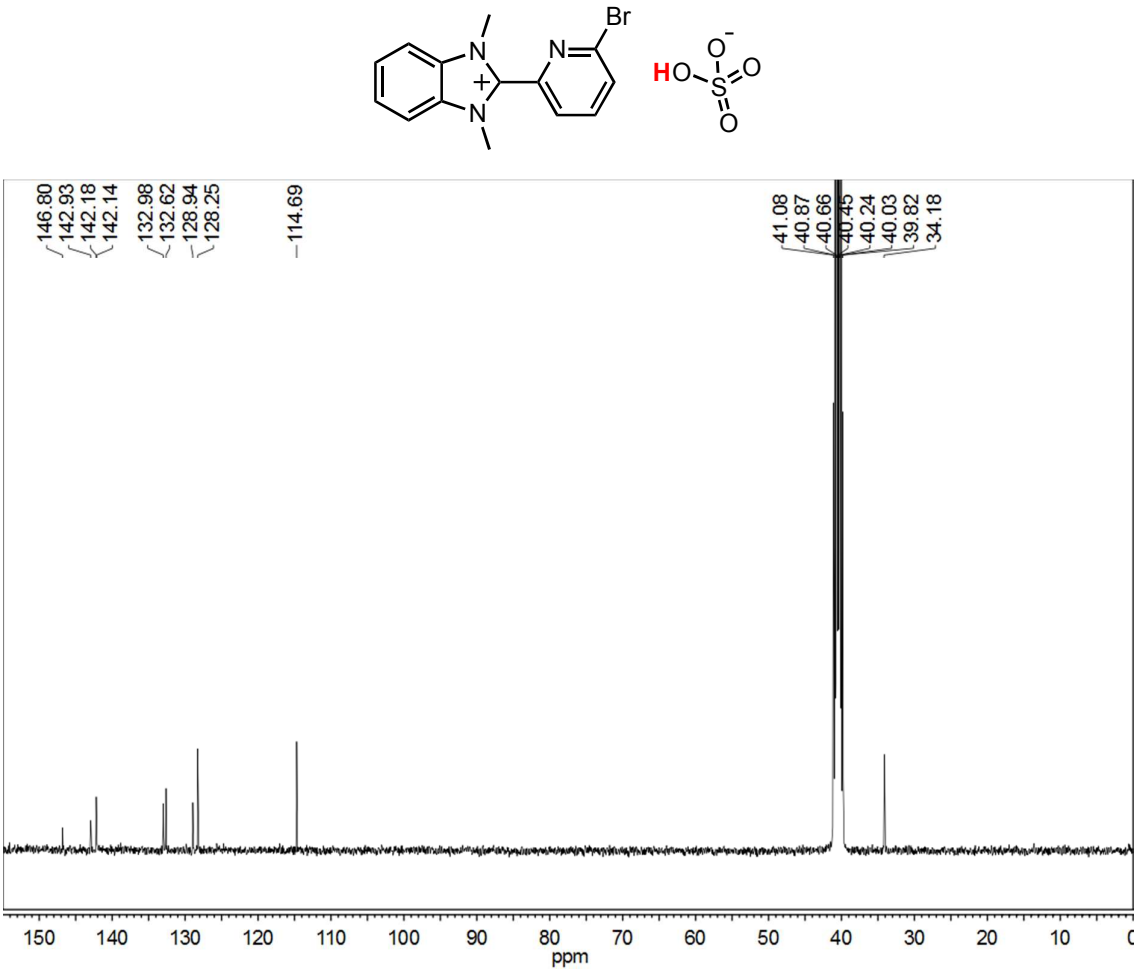
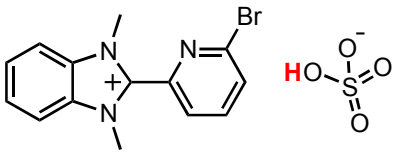
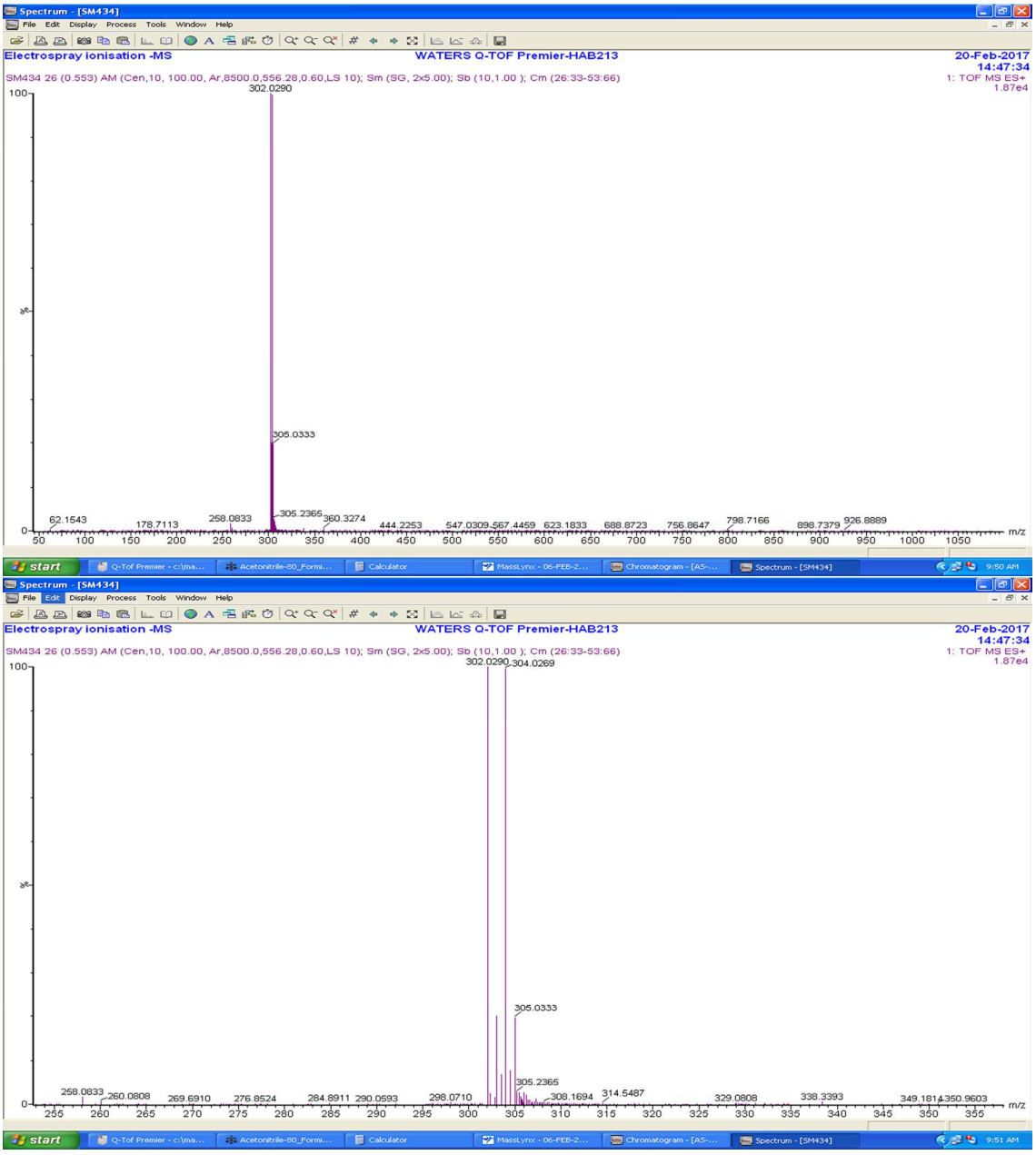


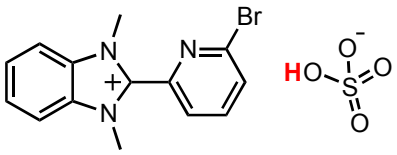
Figure S 29. ESI-MS of [MB3](HSO$_4$) (**cation mode**).

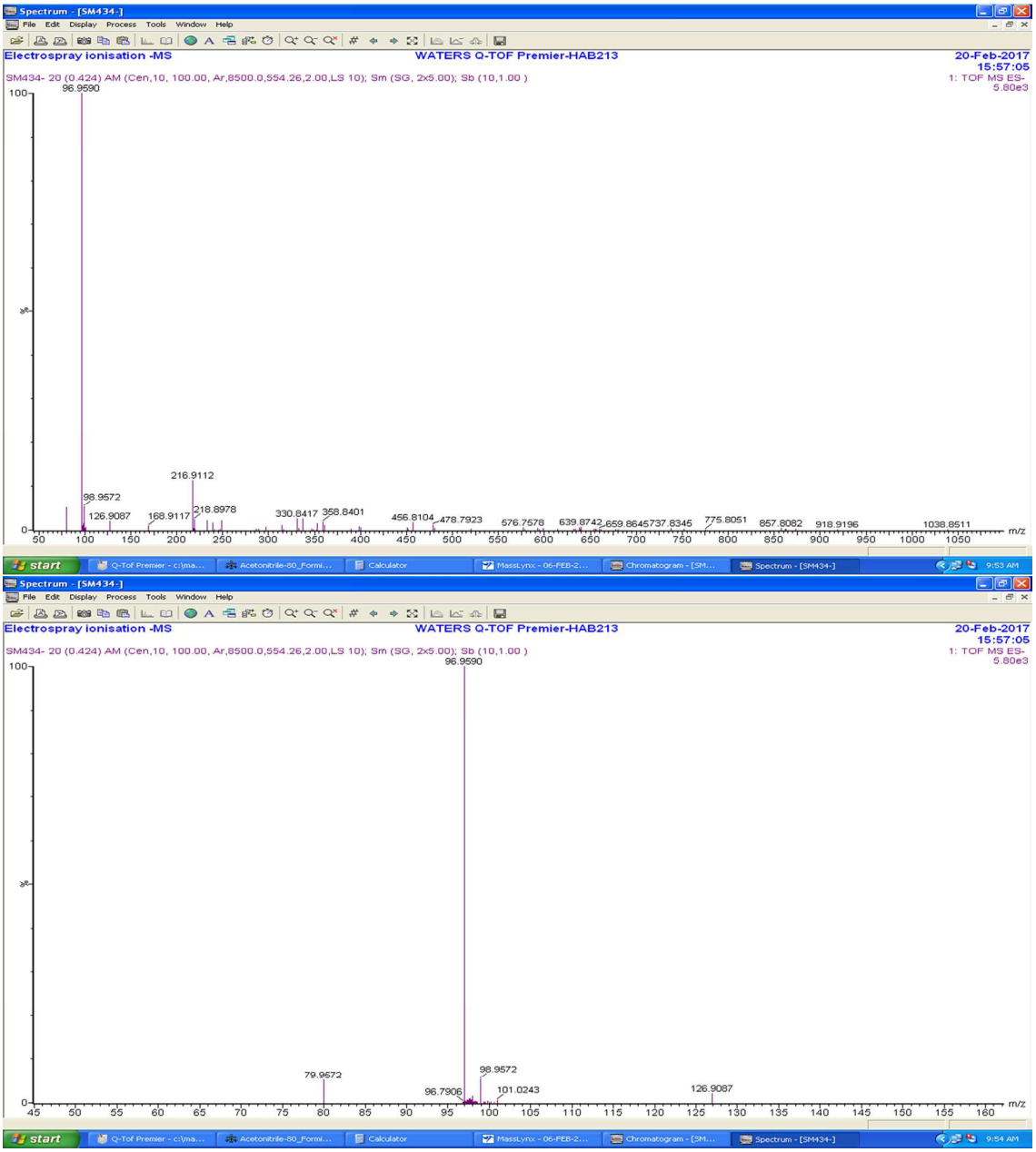




See Figure S 26 for simulated isotopic distribution.

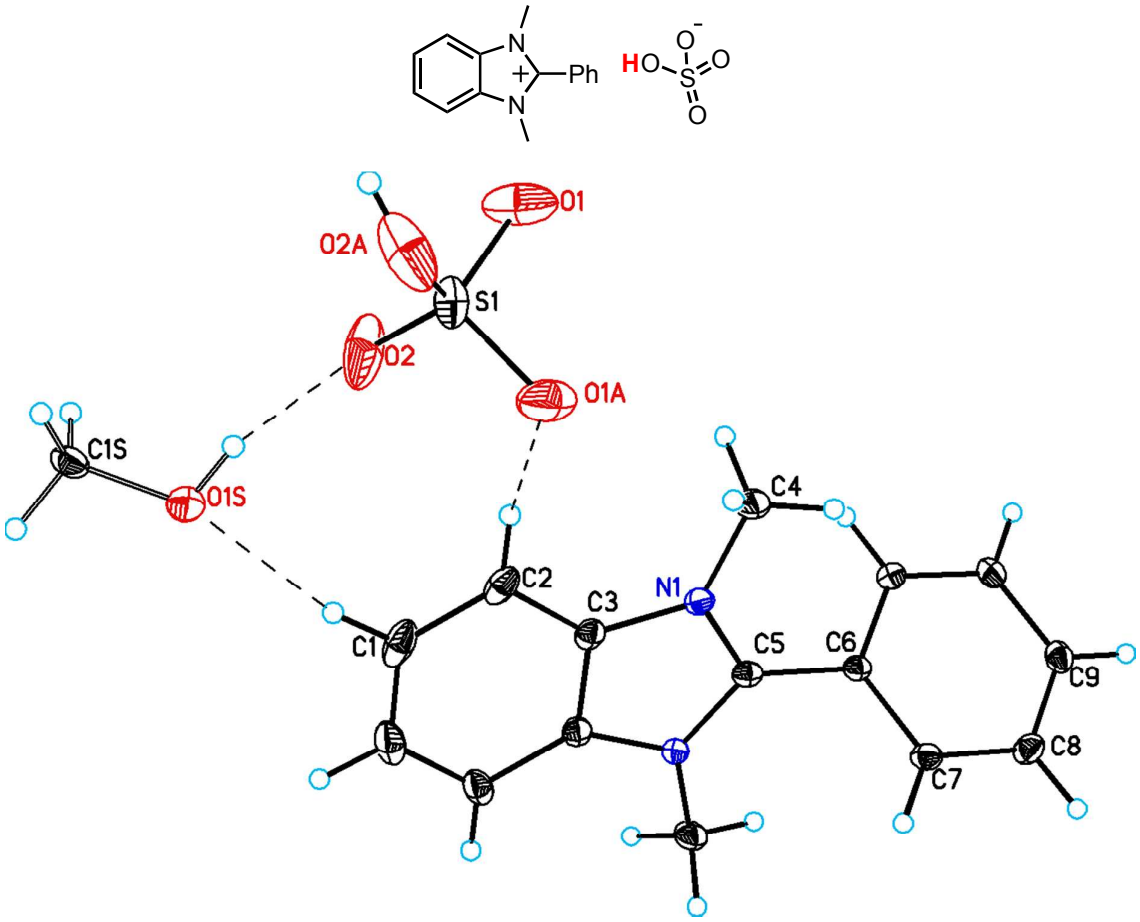
Figure S 30. ESI-MS of [MB3](HSO_4) (**anion mode**).





See Figure S 9 for simulated isotopic distribution.

Figure S 31. Solid state structure of [MB1](HSO$_4$)



Selected distances(Å) and angles(°): S(1)-O(2)#1, 1.408(4); S(1)-O(2), 1.408(4); S(1)-O(2A), 1.477(18);S(1)-O(2A)#1, 1.477(18); S(1)-O(1), 1.495(4); S(1)-O(1)#1, 1.495(4); S(1)-O(1A), 1.528(16); S(1)-O(1A)#1 , 1.528(16); O(2)#1-S(1)-O(2), 116.5(3); O(2)#1-S(1)-O(2A)#1, 50.4(8); O(2)-S(1)-O(2A)#1, 93.4(7); O(2)#1-S(1)-O(1), 106.5(2); O(2)-S(1)-O(1), 111.6(3); O(2)#1-S(1)-O(1)#1, 111.6(3); O(2)-S(1)-O(1)#1, 106.5(2); O(1)-S(1)-O(1)#1, 103.4(3); O(2A)-S(1)-O(1A), 106.1(9); O(2A)#1-S(1)-O(1A) , 13.8(11); O(2)#1-S(1)-O(1A)#1, 73.5(9); O(2)-S(1)-O(1A)#1, 159.5(7); O(1)-S(1)-O(1A)#1, 80.1(8); O(1)#1-S(1)-O(1A)#1, 53.3(7).

Figure S 32. Packing diagram of [MB1](HSO_4)

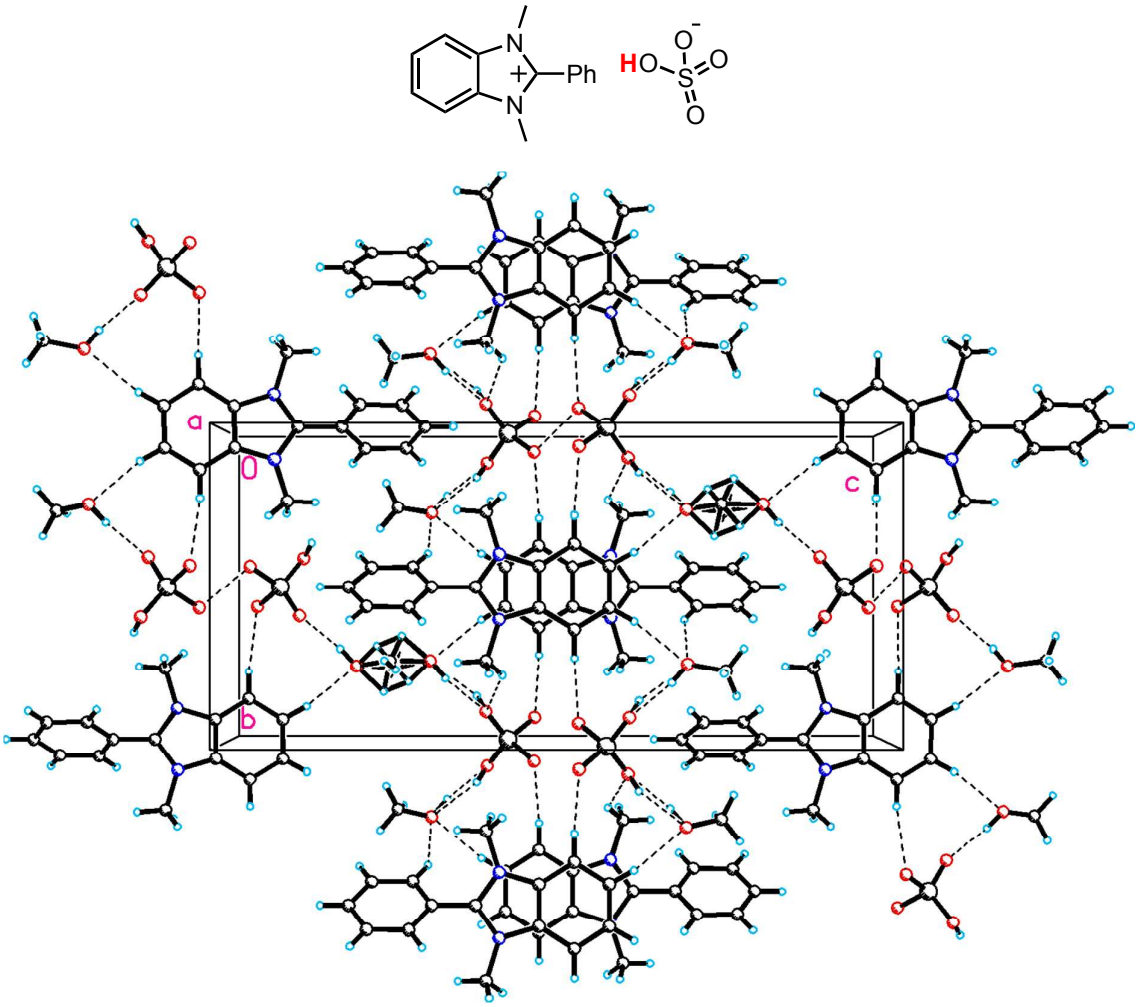
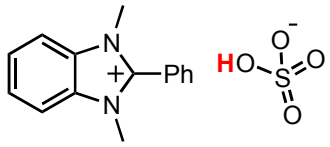
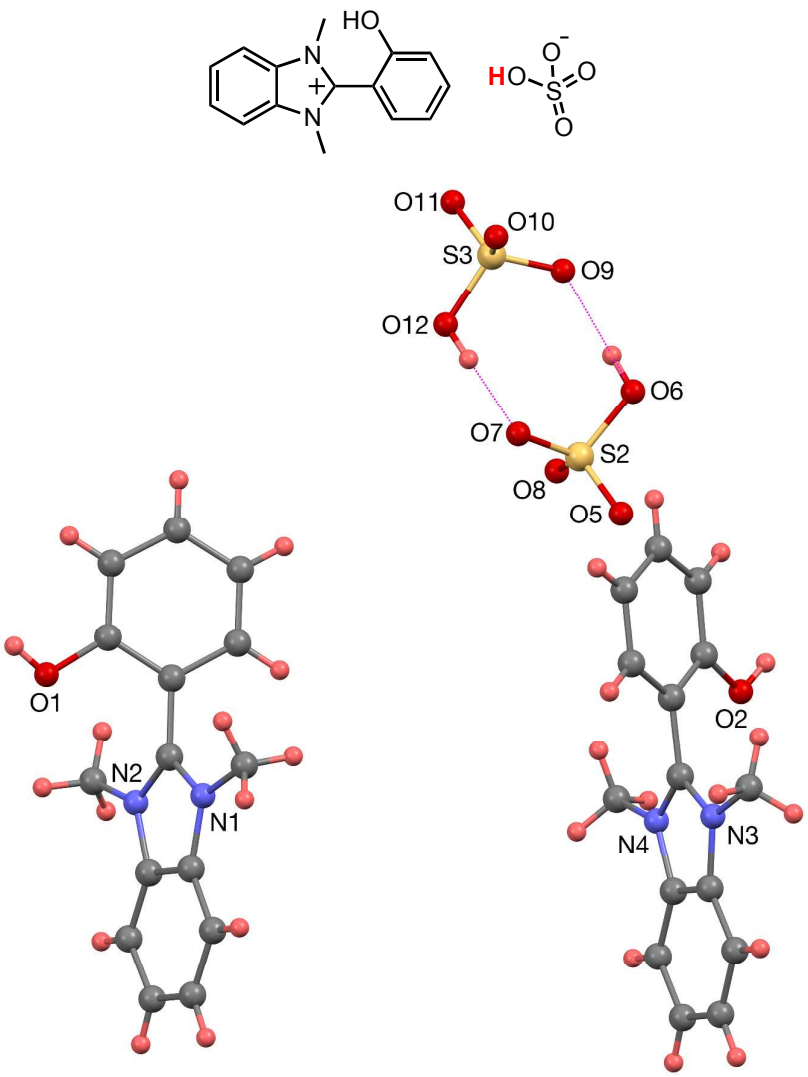


Table S 1. Crystal data for [MB1](HSO$_4$)



Identification code	12janb	CCDC- 1541081
Empirical formula	C16 H20 N2 O5 S	
Formula weight	352.40	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P n n a	
Unit cell dimensions	a = 6.7039(12) Å	α = 90°.
	b = 10.6697(18) Å	β = 90°.
	c = 22.602(4) Å	γ = 90°.
Volume	1616.7(5) Å^3	
Z	4	
Density (calculated)	1.448 Mg/m^3	
Absorption coefficient	0.230 mm^{-1}	
F(000)	744	
Theta range for data collection	3.170 to 25.249°.	
Index ranges	-7<=h<=8, -12<=k<=12, -27<=l<=23	
Reflections collected	11999	
Independent reflections	1466 [R(int) = 0.0789]	
Completeness to theta = 25.249°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7457 and 0.6437	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	1466 / 13 / 130	
Goodness-of-fit on F^2	1.024	
Final R indices [I>2sigma(I)]	R1 = 0.0648, wR2 = 0.1552	
R indices (all data)	R1 = 0.1008, wR2 = 0.1754	
Largest diff. peak and hole	0.782 and -0.376 e.Å^{-3}	

Figure S 33. Solid state structure of [MB2](HSO_4)



Selected distances(Å) and angles(°): S(2)-O(8) 1.4315(16); S(2)-O(5), 1.4385(16); S(2)-O(7), 1.4761(15); S(2)-O(6), 1.5574(16); S(3)-O(11), 1.4452(16); S(3)-O(10), 1.4502(16); S(3)-O(9), 1.4640(15); S(3)-O(12), 1.5521(15); O(1)-C(11), 1.352(3); O(2)-C(26), 1.348(2); N(4)-C(17), 1.338(3); N(4)-C(19), 1.390(3); N(4)-C(18), 1.460(3);N(3)-C(17), 1.341(3); N(3)-C(20), 1.396(3); N(3)-C(16), 1.468(3); N(2)-C(2), 1.341(3); N(2)-C(4), 1.388(3); N(2)-C(3), 1.467(3); N(1)-C(2), 1.336(3); N(1)-C(5), 1.395(3); N(1)-C(1), 1.469(3); O(8)-S(2)-O(5), 115.23(10); O(8)-S(2)-O(7), 111.48(10); O(5)-S(2)-O(7), 111.59(9); O(8)-S(2)-O(6), 107.90(10); O(5)-S(2)-O(6), 103.69(9); O(7)-S(2)-O(6), 106.17(9); O(11)-S(3)-O(10), 113.61(9); O(11)-S(3)-O(9), 111.64(9); O(10)-S(3)-O(9), 112.04(9); O(11)-S(3)-O(12), 104.15(9); O(10)-S(3)-O(12), 107.48(9); O(9)-S(3)-O(12), 107.33(9); C(17)-N(4)-C(19), 108.69(18); C(17)-N(4)-C(18), 126.17(18); C(19)-N(4)-C(18), 125.10(18); C(17)-N(3)-C(20), 108.57(17); C(17)-N(3)-C(16), 126.32(18); C(20)-N(3)-C(16), 125.06(18); C(2)-N(2)-C(4), 108.71(17); C(2)-N(2)-C(3), 125.82(18); C(4)-N(2)-C(3), 125.46(18); C(2)-N(1)-C(5), 108.90(17); C(2)-N(1)-C(1), 125.76(18); C(5)-N(1)-C(1), 125.33(18).

Figure S 34. Packing diagram of [MB2](HSO$_4$)

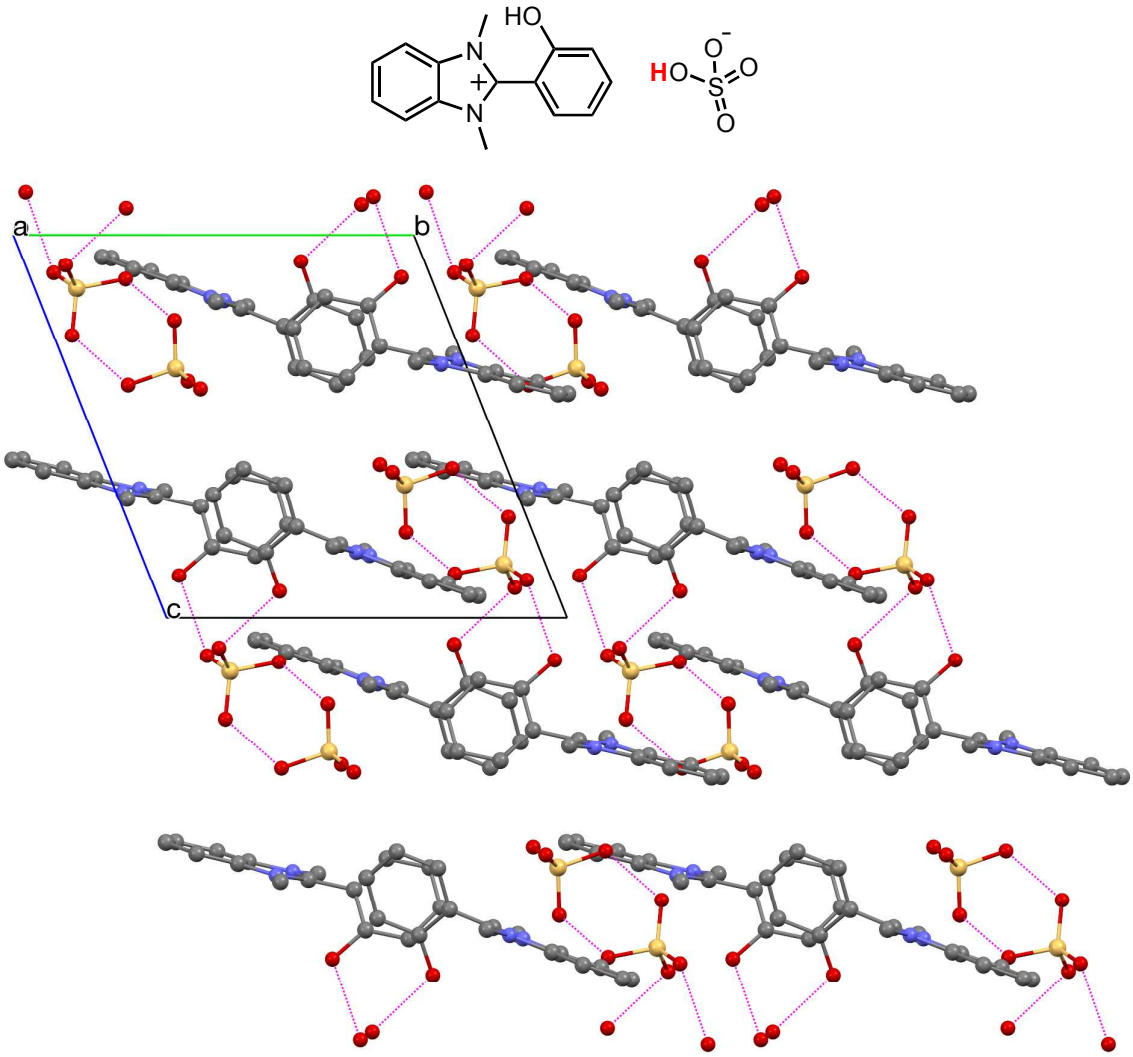
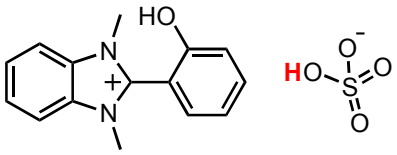
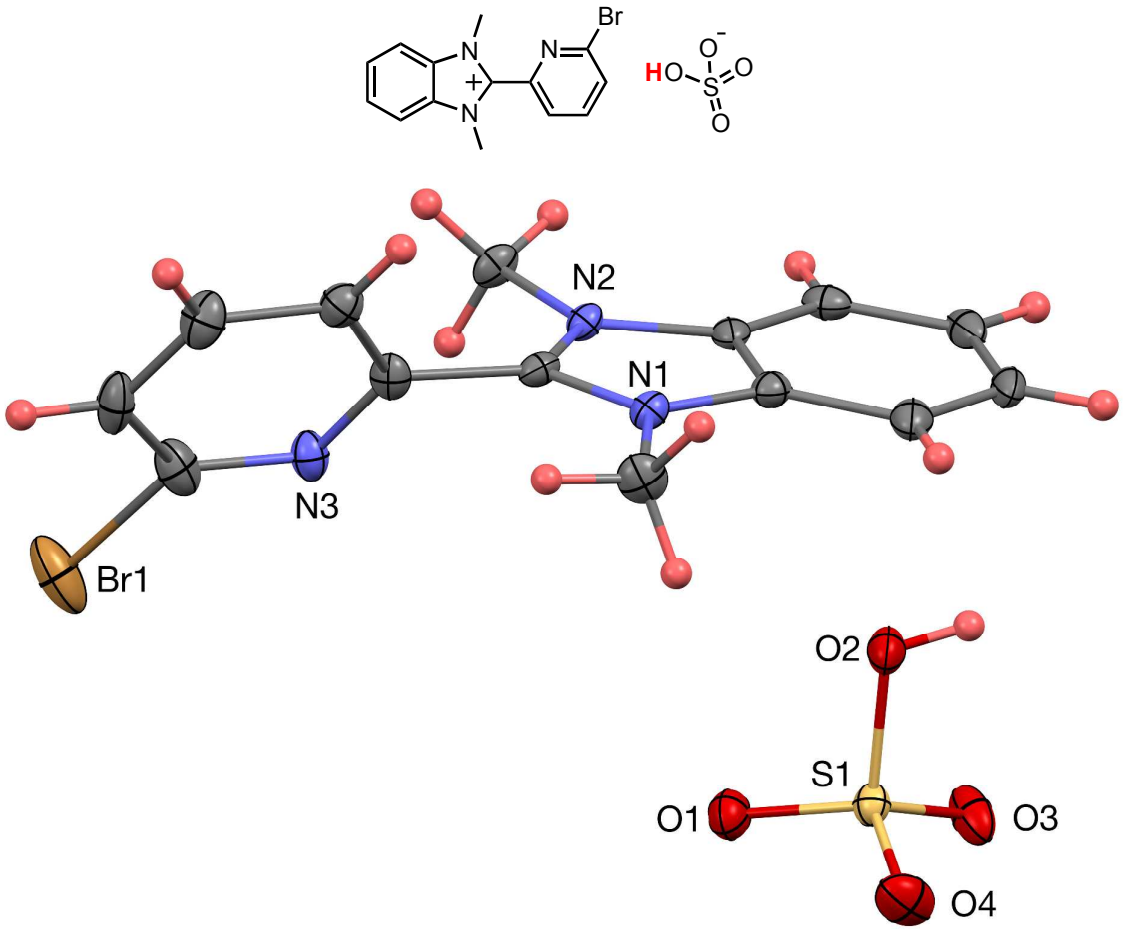


Table S 2. Crystal data for [MB2](HSO_4)



Identification code	6nova_0m	CCDC- 1541079
Empirical formula	C15 H16 N2 O5 S	
Formula weight	336.36	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 10.8743(8) Å	α = 64.0149(19)°.
	b = 12.9713(9) Å	β = 71.014(2)°.
	c = 13.3718(10) Å	γ = 70.772(2)°.
Volume	1564.1(2) $Å^3$	
Z	4	
Density (calculated)	1.428 Mg/m^3	
Absorption coefficient	0.234 mm^{-1}	
F(000)	704	
Crystal size	0.22 x 0.20 x 0.18 mm^3	
Theta range for data collection	1.98 to 26.00°.	
Index ranges	-13<=h<=13, -15<=k<=15, -16<=l<=16	
Reflections collected	20542	
Independent reflections	6143 [R(int) = 0.0441]	
Completeness to theta = 26.00°	99.9 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6143 / 0 / 423	
Goodness-of-fit on F^2	1.020	
Final R indices [I>2sigma(I)]	R1 = 0.0439, wR2 = 0.1279	
R indices (all data)	R1 = 0.0619, wR2 = 0.1427	
Largest diff. peak and hole	0.445 and -0.557 $e.Å^{-3}$	

Figure S 35. Solid state structure of [MB3](HSO_4)



Selected distances(Å) and angles(°): Br(1)-C(14) 1.896(4); S(1)-O(4), 1.431(3); S(1)-O(1), 1.436(3); S(1)-O(3), 1.468(3); S(1)-O(2), 1.576(3); N(2)-C(2), 1.340(4); N(2)-C(4), 1.393(4); N(2)-C(1), 1.467(4); N(1)-C(2), 1.344(4); N(1)-C(9), 1.389(4); N(1)-C(3), 1.462(5); N(3)-C(14), 1.308(5); N(3)-C(10), 1.333(5); O(4)-S(1)-O(1), 114.54(17); O(4)-S(1)-O(3), 112.36(17); O(1)-S(1)-O(3), 110.75(17); O(4)-S(1)-O(2), 107.74(16); O(1)-S(1)-O(2), 105.14(14); O(3)-S(1)-O(2), 105.59(15); C(2)-N(2)-C(4), 108.7(3); C(2)-N(2)-C(1), 125.5(3); C(4)-N(2)-C(1), 125.8(3); C(2)-N(1)-C(9), 108.3(3); C(2)-N(1)-C(3), 125.9(3); C(9)-N(1)-C(3), 125.6(3); C(14)-N(3)-C(10), 116.4(3); C(4)-C(9)-N(1), 107.2(3); C(4)-C(9)-C(8), 122.2(3); N(1)-C(9)-C(8), 130.6(3); N(2)-C(2)-N(1), 109.4(3); N(2)-C(2)-C(10), 124.8(3); N(1)-C(2)-C(10), 125.7(3); N(3)-C(14)-Br(1), 117.4(3); C(13)-C(14)-Br(1), 117.4(3).

Figure S 36. Packing diagram of [MB3](HSO_4)

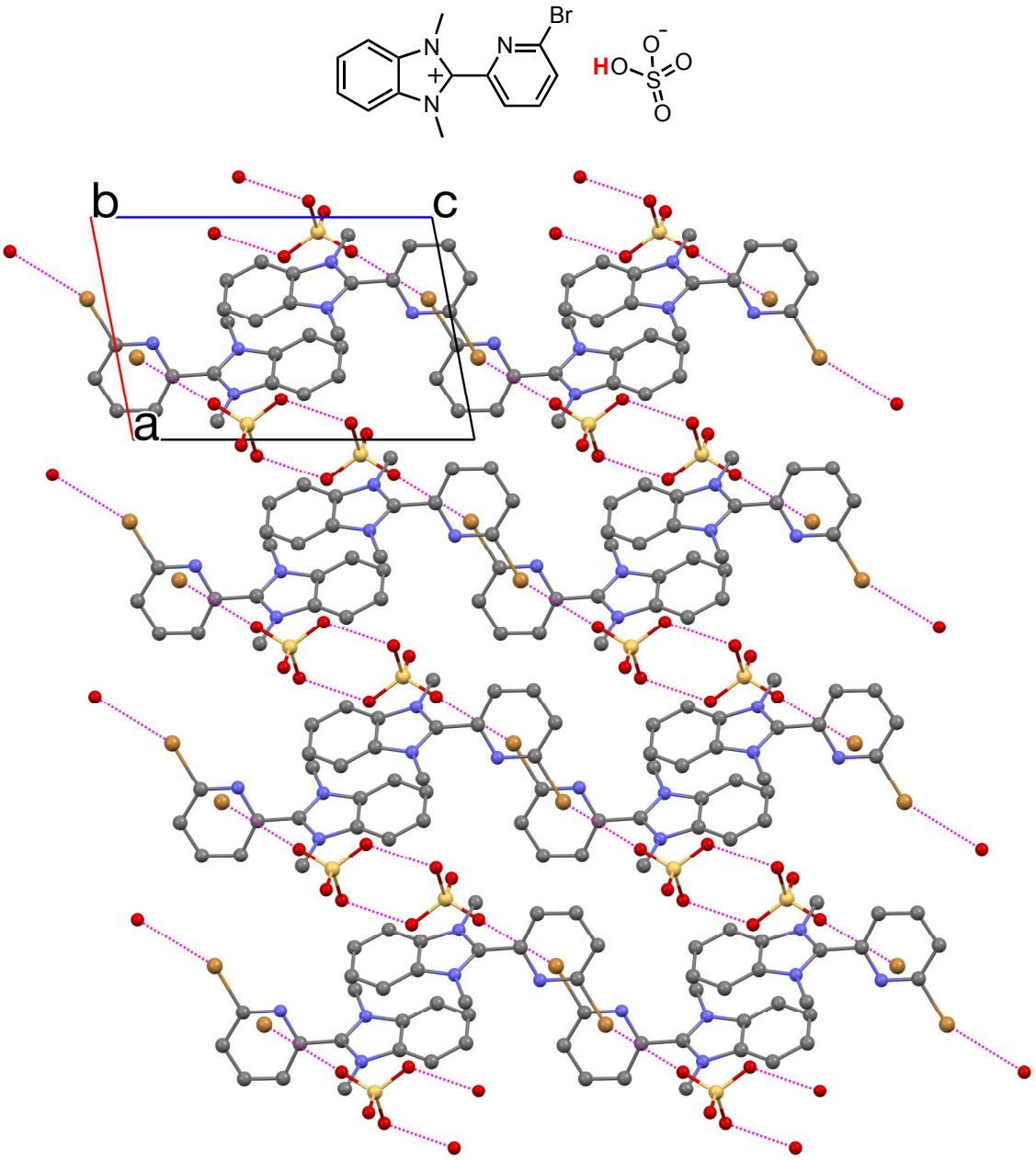
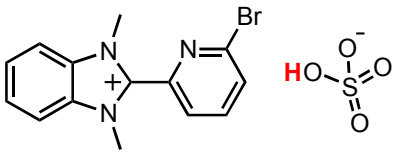


Table S 3. Crystal data for [MB3](HSO$_4$).



Identification code	20febc_0m	CCDC-1541080
Empirical formula	C14 H14 Br N3 O4 S	
Formula weight	400.25	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 7.907(5) Å	α = 83.119(5)°.
	b = 8.536(5) Å	β = 78.561(5)°.
	c = 11.911(5) Å	γ = 83.567(5)°.
Volume	779.1(7) Å^3	
Z	2	
Density (calculated)	1.706 Mg/m^3	
Absorption coefficient	2.796 mm^{-1}	
F(000)	404	
Crystal size	0.20 x 0.18 x 0.16 mm^3	
Theta range for data collection	2.64 to 25.06°.	
Index ranges	-9<=h<=9, -10<=k<=10, -14<=l<=14	
Reflections collected	9504	
Independent reflections	2754 [R(int) = 0.0343]	
Completeness to theta = 25.06°	99.6 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2754 / 0 / 211	
Goodness-of-fit on F^2	1.249	
Final R indices [I>2sigma(I)]	R1 = 0.0428, wR2 = 0.1456	
R indices (all data)	R1 = 0.0495, wR2 = 0.1523	
Largest diff. peak and hole	2.029 and -1.349 e.Å^{-3}	

References:

1. X. Q. Zhu, M. T. Zhang, A. Yu, C. H. Wang and J. P. Cheng, *J. Am. Chem. Soc.*, 2008, 130, 2501.
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3. T. Igarashi, E. Tayama, H. Iwamoto and E. Hasegawa, *Tetrahedron Lett.*, 2013, 54, 6874.
4. MestReNova, Mestrelab Research S.L., Santiago de Compostela, Spain, www.mestrelab.com, 2014.
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