Electronic Supplementary Material (ESI) for RSC Advances.

The Curious Case of Salicylidene based Fluoride Sensors: Chemosensors or Chemodosimeters or None of Them

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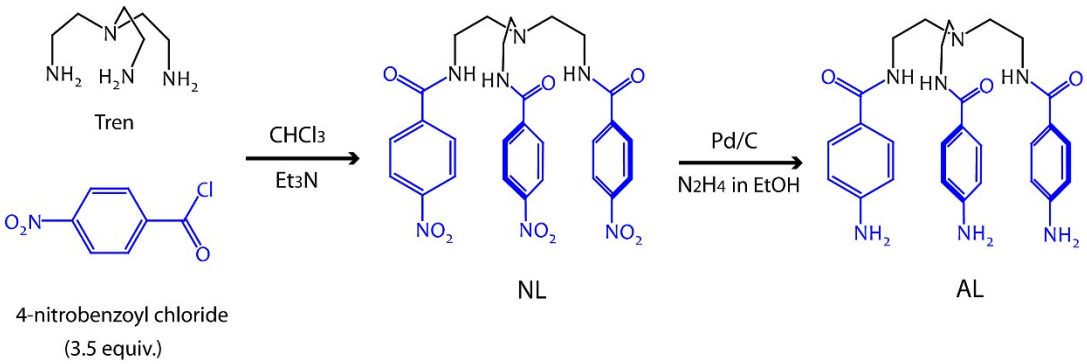
1. Synthesis and Characterization of Salicylidene Schiff Base Compounds SL and CL1-3

(1A) Synthesis of tris(4-amino-N-ethylbenzamide)amine (AL): Tris(4-amino-N-ethylbenzamide)amine (**AL**) was synthesized by reduction of its nitro analogue (Tris(4-nitro-N-ethylbenzamide)amine**, NL**) which was synthesized by modification of the reported literature procedure (Scheme S1).[1] **NL** was synthesized by the reaction of tris(2-aminoethyl)amine, (Tren) with 4-nitrobenzoyl chloride in 1 : 3.5 molar ratio at room temperature in dry chloroform. In a 100 mL flat bottom flask, 0.73 mL (5 mmol) of tris(2-aminoethyl)amine was dissolved in 25 mL of chloroform and 3.5 g of 4-nitrobenzoyl chloride (17.5 mmol) was added in portions into the above solution with constant stirring at room temperature. The reaction mixture was allowed to stir overnight at room temperature followed by the addition of 3 ml (excess) triethylamine and stirred for another 1 hrs. Reaction of tren with 4-nitrobenzoyl chloride generates HCl in the reaction medium, which eventually protonate the tertiary nitrogen of the formed **NL**. Triethylamine was added to basify the reaction mixture so that **NL** can be obtained in its neutral form. The precipitate obtained was then filtered, collected in a 250 ml flat bottom flask and washed with 50 ml of methanol in the presence of 1 ml of triethylamine under stirring. The compound was finally filtered again and washed with another 50 ml of methanol over the filter paper to ensure its purity for subsequent reduction reaction.

In a 250 ml flat bottom flask, 1 g of **NL** was dispersed in 100 ml of ethanol and 100 mg of Pd/C and 1 ml of hydrazine hydrate was added in to the flask. The reaction mixture was then refluxed overnight at about 80 ˚C and filtered to remove the heterogeneous Pd/C catalyst. The filtrate was then allowed to evaporate in a beaker at room temperature when colorless crystals of **AL** were obtained in quantitative yield within 2 days. The crystals were collected by decantation/filtration and washed with 10 ml of ethanol to ensure its purity for spectroscopy analysis. The compound was characterized by NMR and FT-IR spectroscopy.

Isolated yield of **AL**: 614 mg (percentage yield 72%). The compound is highly soluble in dimethylformamide, and dimethyl sulfoxide, soluble in methanol/ethanol on heating, and insoluble in tetrahydrofuran, chloroform and acetonitrile.

Characterization of **AL**: ^{1}H-NMR (400 MHz, DMSO-d_6) chemical shift in δ ppm: 2.50 (DMSO-CH_3), 2.64 (t, 6xN**CH_2**), 3.30 (t, 6xNCH$_2$**CH_2**), 3.37 (HOD), 5.56 (s, 3x**NH_2**), 6.50 (d, 6xCH), 7.55 (d, 6xCH), 7.94 (t, 3x**NH**).



Scheme S1: Synthesis of AL from tris(2-aminoethylamine) and 4-nitrobenzoyl chloride.

(1B) Synthesis of tris-4-(2-hydroxybenzylideneamino)-N-(2-aminoethyl)benzamide (SL): Salicylidene based tripodal amide receptor **SL** was synthesized by Schiff base condensation reaction of **AL** with salicylaldehyde in methanol under reflux (Scheme 1, main manuscript). In a 250 ml flat bottom flask, 500 mg of **AL** (1.0 mmol) and 400 mg (350 μL) of 2-hydroxy benzaldehyde (3.5 mmol) were mixed in 100 ml of methanol. After overnight refluxing of the reaction mixture at 60 °C, the yellow precipitate formed was filtered and washed with 20 ml of methanol to ensure its purity for spectroscopy analysis. The compound was characterized by NMR, and FT-IR spectroscopy**.**

Isolated yield of **SL**: 550 mg (percentage yield 67%). The compound is highly soluble in dimethylformamide, and dimethyl sulfoxide, soluble in methanol/ethanol on heating, and insoluble in tetrahydrofuran, chloroform and acetonitrile.

Characterization of **SL**: **^{1}H-NMR** (400 MHz, DMSO-d_6) chemical shift in δ ppm: 2.75 (3xN**CH_2**), 3.42 (3xNCH$_2$**CH_2**), 6.95 (6xCH), 7.42 (9xCH), 7.57 (3xCH), 7.88 (6xCH), 8.40 (3xNH), 8.94 (3x**CH**=N), 12.84 (3x**OH**). **^{13}C-NMR** (100 MHz, DMSO-d_6) chemical shift in δ ppm: 31.16 (3xCH$_3$OH), 38.10 (3x-NCH$_2$), 53.63 (3x-NCH$_2$CH$_2$), 117.07 (3x-CH), 119.63 (3x-CH), 119.68 (3x-CH), 121.60 (3x-CH), 128.94 (3x-CH), 132.98 (3x-CH), 133.06 (3x-CH), 134.05 (3x-CH), 150.81 (3x-CH), 160.75 (3x-CH), 164.67 (3xC=N), 166.16 (3xC=O). HR-MS m/z 816.350 (SL+H$^+$)

(1C) Synthesis of 4-(2-hydroxybenzylideneamino)benzonitrile (CL1): 1 g of 4-aminobenzonitrile (8.5 mmol) was dissolved in 25 ml of methanol and 1.25 g (1.06 ml) of 2-hydroxybenzaldehyde (10.15 mmol) was added into the solution. The solution mixture was then stirred for about 12 hrs. and the yellow precipitate formed was then filtered and washed with 15 ml (3 x 5 ml) of methanol to obtain **CL1**. The compound was then air dried at room temperature and characterized by ^{1}H-NMR, ^{13}C NMR and FT-IR spectroscopy. Isolated yield of **CL1**: 1.5 g (percentage yield 85%). The compound is soluble in dimethylformamide, dimethyl sulfoxide, chloroform and tetrahydrofuran soluble in methanol/ethanol on heating.

^{1}H-NMR (400 MHz, DMSO-d_6) chemical shift in δ ppm: 2.50 (DMSO-CH$_3$), 3.35 (HOD), 7.00 (m, 2xCH), 7.46 (t, 1xCH), 7.54 (d, 2xCH), 7.70 (d, 1xCH), 7.91 (d, 2xN=CH), 12.43 (s, 1xOH). **^{13}C-NMR** (100 MHz, DMSO-d_6) δ ppm: 108.83, 116.72, 118.79, 119.30, 119.35, 122.46, 132.54, 133.64, 134.09, 152.57, 160.24, 165.39. HR-MS m/z 223.086 (CL1+H$^+$)

(1D) Synthesis of 1,2-(2-hydroxybenzylideneamino)benzene (CL2): 1 g of 1,2-phenylenediamine (9.25 mmol) was dissolved in 25 ml of methanol and 1.35 g (1.15 ml) of 2-hydroxybenzaldehyde (11.10 mmol) was added into the solution. The solution mixture was then stirred for about 12 hrs. and the yellow precipitate formed was then filtered and washed with 15 ml (3 x 5 ml) of methanol to obtain **CL2**. The compound was then air dried at room temperature and characterized by ^{1}H-NMR, ^{13}C NMR and FT-IR spectroscopy. Isolated yield of **CL2**: 2.15 mg (percentage yield 81%). The compound is soluble in dimethylformamide, dimethyl sulfoxide, chloroform and tetrahydrofuran.

^{1}H-NMR (400 MHz, DMSO-d_6) chemical shift in δ ppm: 2.51 (DMSO-CH_3), 3.35 (HOD), 6.98 (m, 4xCH), 7.42 (m, 6xCH), 7.68 (d, 2xCH), 8.94 (s, 2xN=CH), 12.95 (s, 2xOH). **^{13}C-NMR** (100 MHz, DMSO-d_6) δ ppm: 116.65, 119.05, 119.47, 119.72, 127.78, 132.44, 133.41, 142.24, 160.37, 164.01. HR-MS m/z 317.128 (CL2+H^+)

(1E) Synthesis of 1,3-(2-hydroxybenzylideneamino)benzene (CL3): 1 g of 1,3-phenylenediamine (9.25 mmol) was dissolved in 25 ml of methanol and 1.35 g (1.15 ml) of 2-hydroxybenzaldehyde (11.10 mmol) was added into the solution. The solution mixture was then stirred for about 12 hrs. and the yellow precipitate formed was then filtered and washed with 15 ml (3 x 5 ml) of methanol to obtain **CL3**. The compound was then air dried at room temperature and characterized by ^{1}H-NMR, ^{13}C NMR and FT-IR spectroscopy. Isolated yield of **CL3**: 1.95 mg (percentage yield 74%). The compound is soluble in dimethylformamide, dimethyl sulfoxide, chloroform and tetrahydrofuran.

^{1}H-NMR (400 MHz, DMSO-d_6) chemical shift in δ ppm: 2.51 (DMSO-CH_3), 3.35 (HOD), 7.35 (m, 4xCH), 7.42 (d, 2xCH), 7.47 (m, 2xCH), 7.57 (m, 2xCH), 7.69 (d, 2xCH), 9.06 (s, 2xN=CH), 13.02 (s, 2xOH). **^{13}C-NMR** (100 MHz, DMSO-d_6) δ ppm: 113.85, 116.64, 119.18, 119.26, 120.15, 130.33, 132.63, 133.44, 149.26, 160.32, 164.12. HR-MS m/z 317.128 (CL3+H^+)

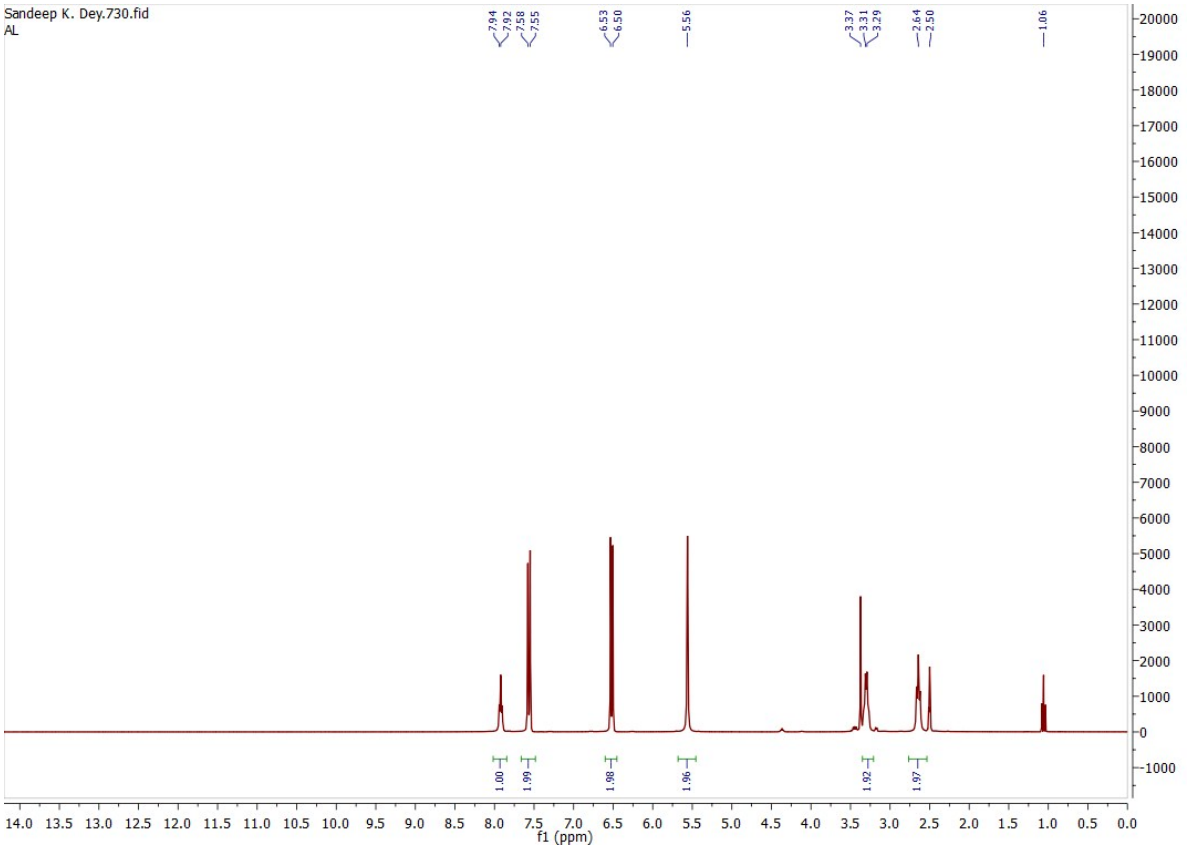


Fig. S1: ^{1}H-NMR spectrum of **AL** in DMSO-d_6.

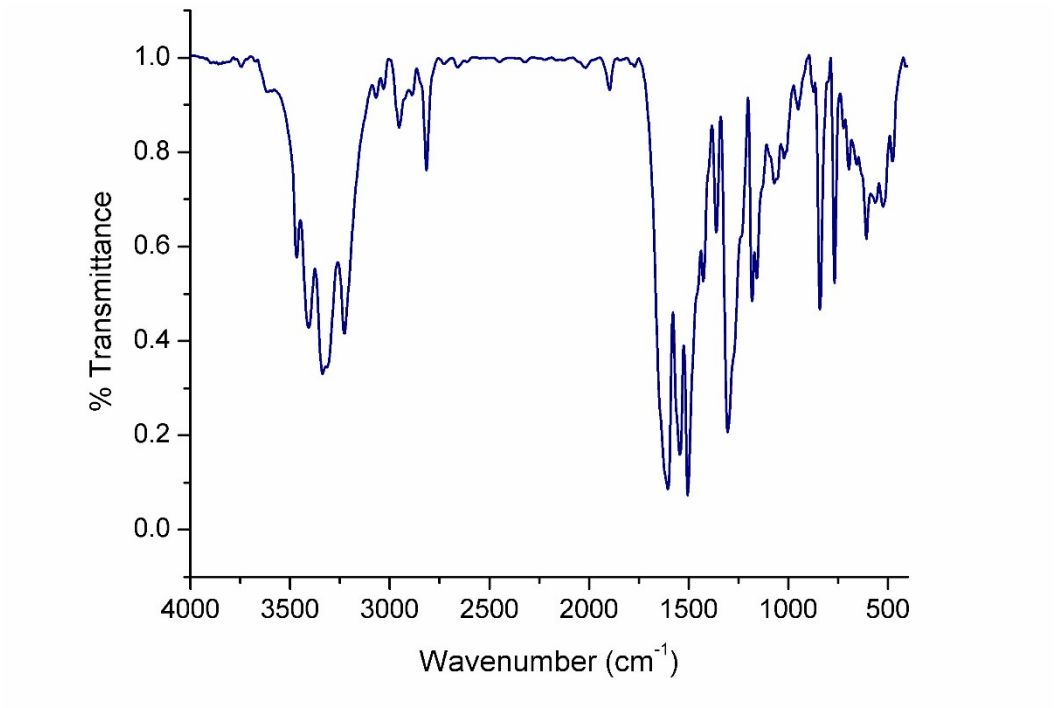


Fig. S2: FT-IR spectrum of **AL** (KBr).

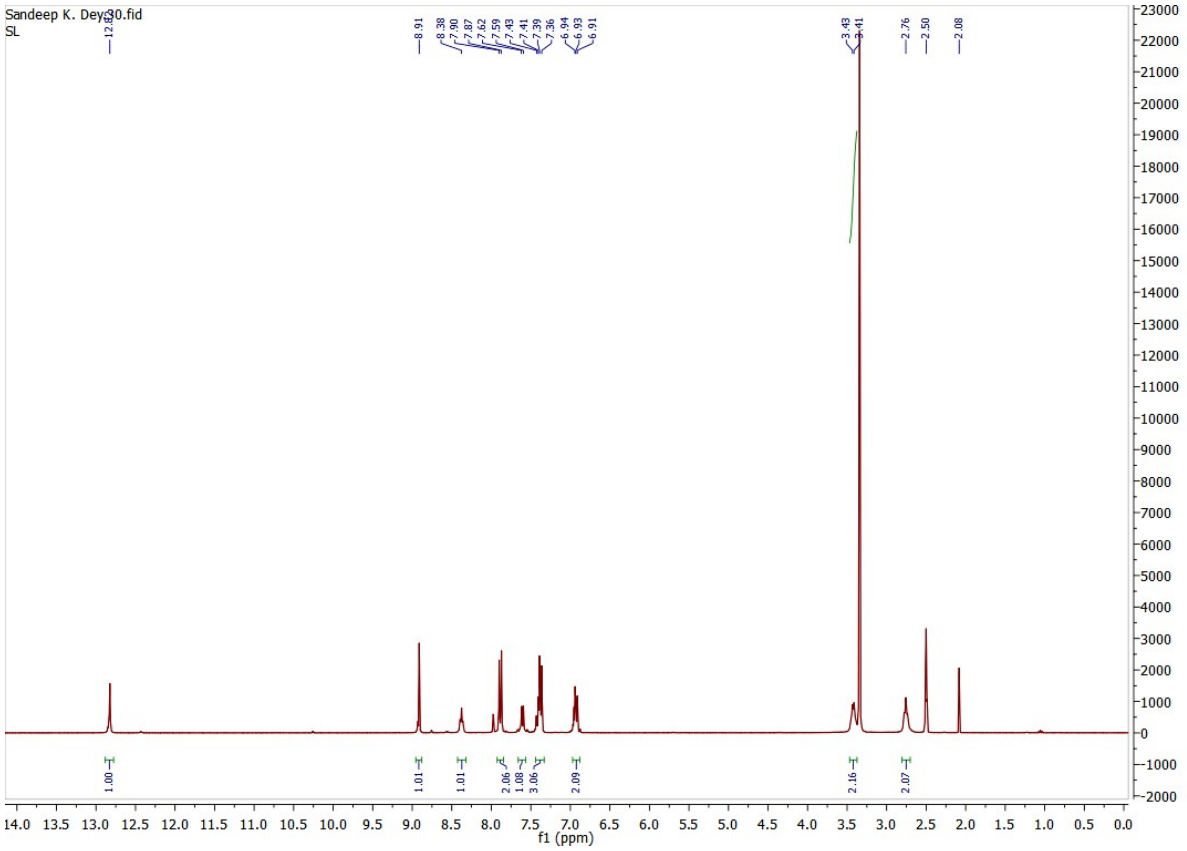


Fig. S3: ^{1}H-NMR spectrum of **SL** in DMSO-d_6.

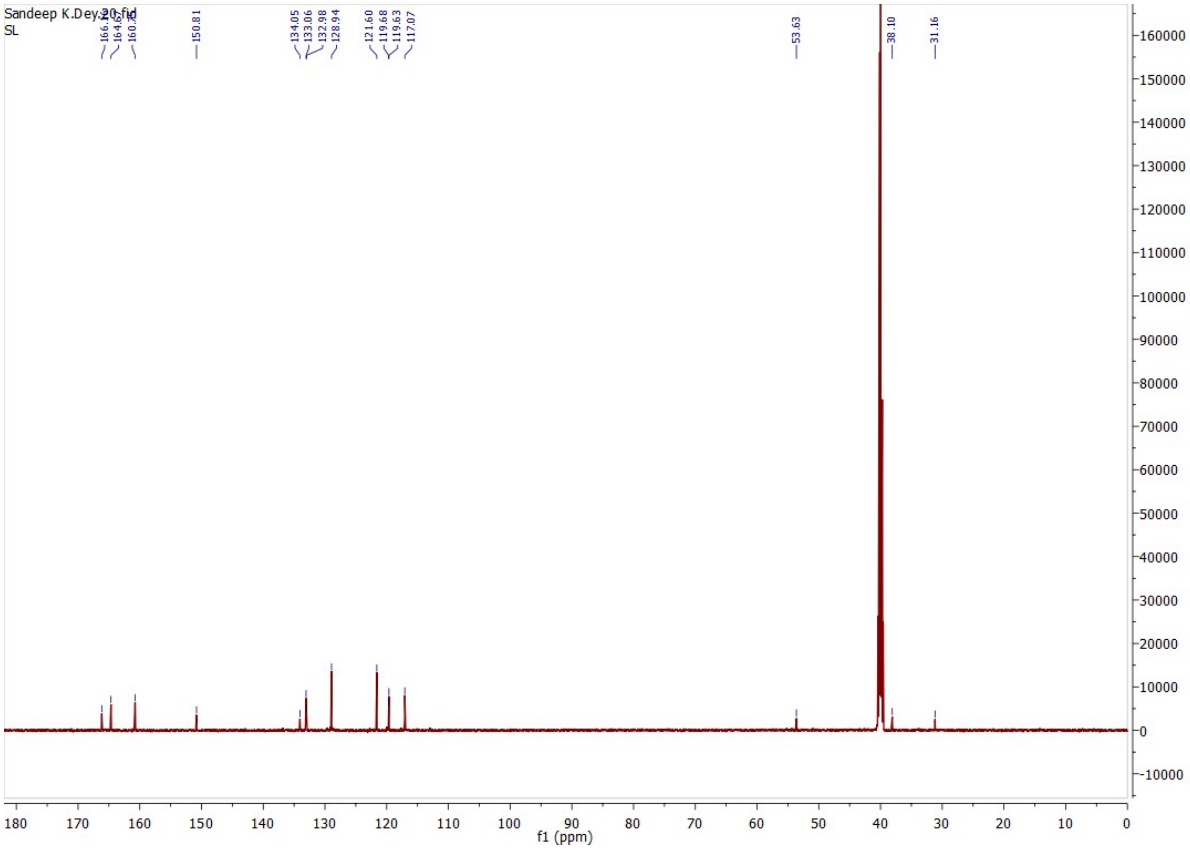


Fig. S4: ^{13}C-NMR spectrum of **SL** in DMSO-d_6 from 0-180 ppm (full spectrum).

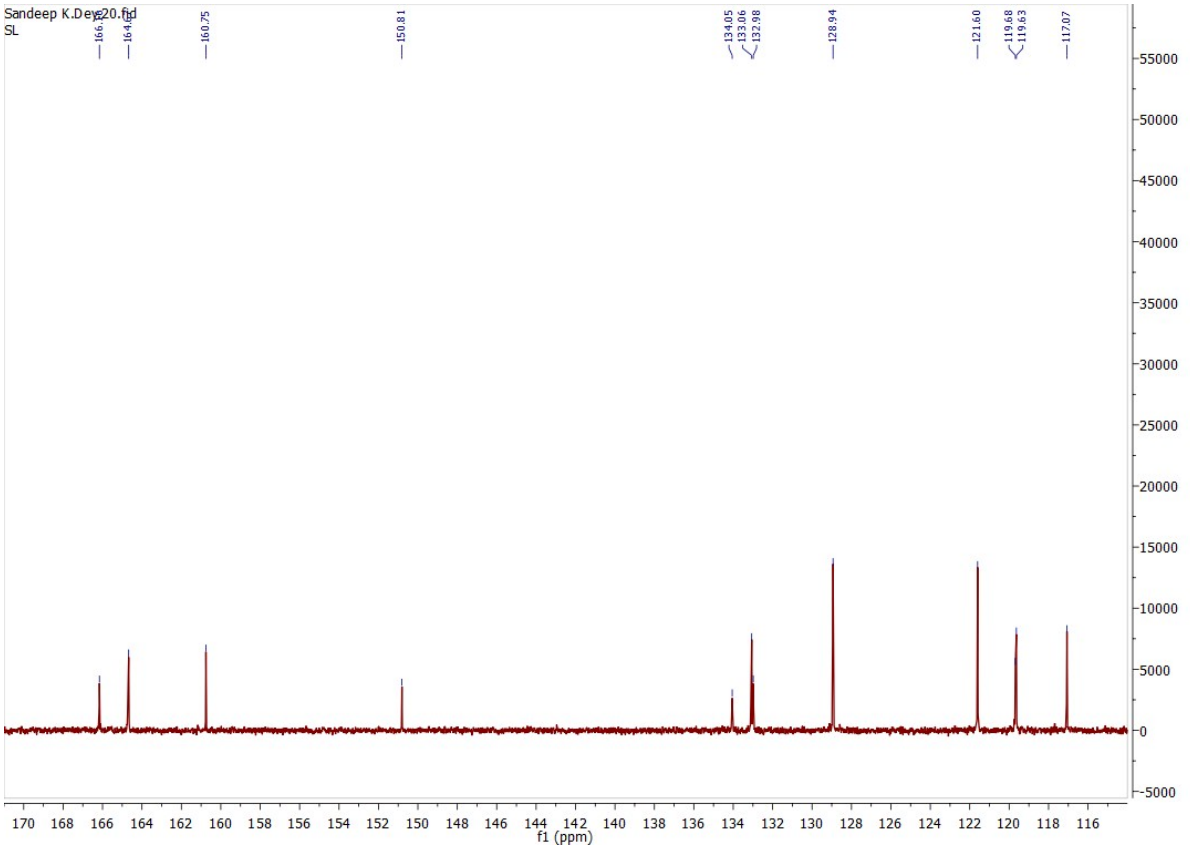


Fig. S5: ^{13}C-NMR spectrum of **SL** in DMSO-d_6 from 114-171 ppm (aromatic region).

Mass Spectrum SmartFormula Report

Analysis Info

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Method	tune_low_new.m	Operator	Peter Tommes
Sample Name	S. Dey SL in Aceton (CH3OH)	Instrument	maXis 288882.20213
Comment			

Acquisition Parameter

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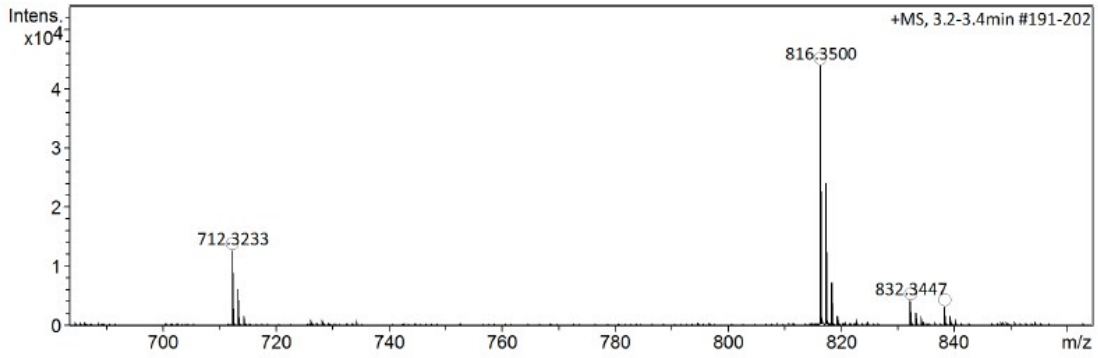

Fig. S6: ESI HR-MS of **SL** in acetone. Peak at m/z 816.35 corresponds to (**SL**+H^+).

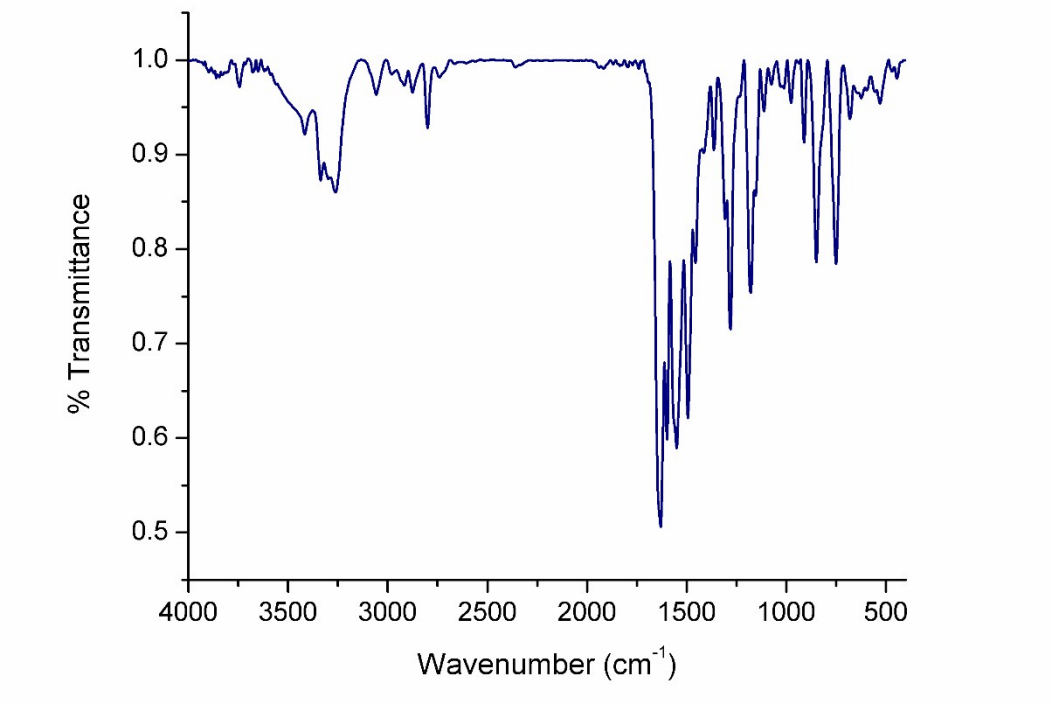

Fig. S7: FT-IR spectrum of **SL** (KBr).

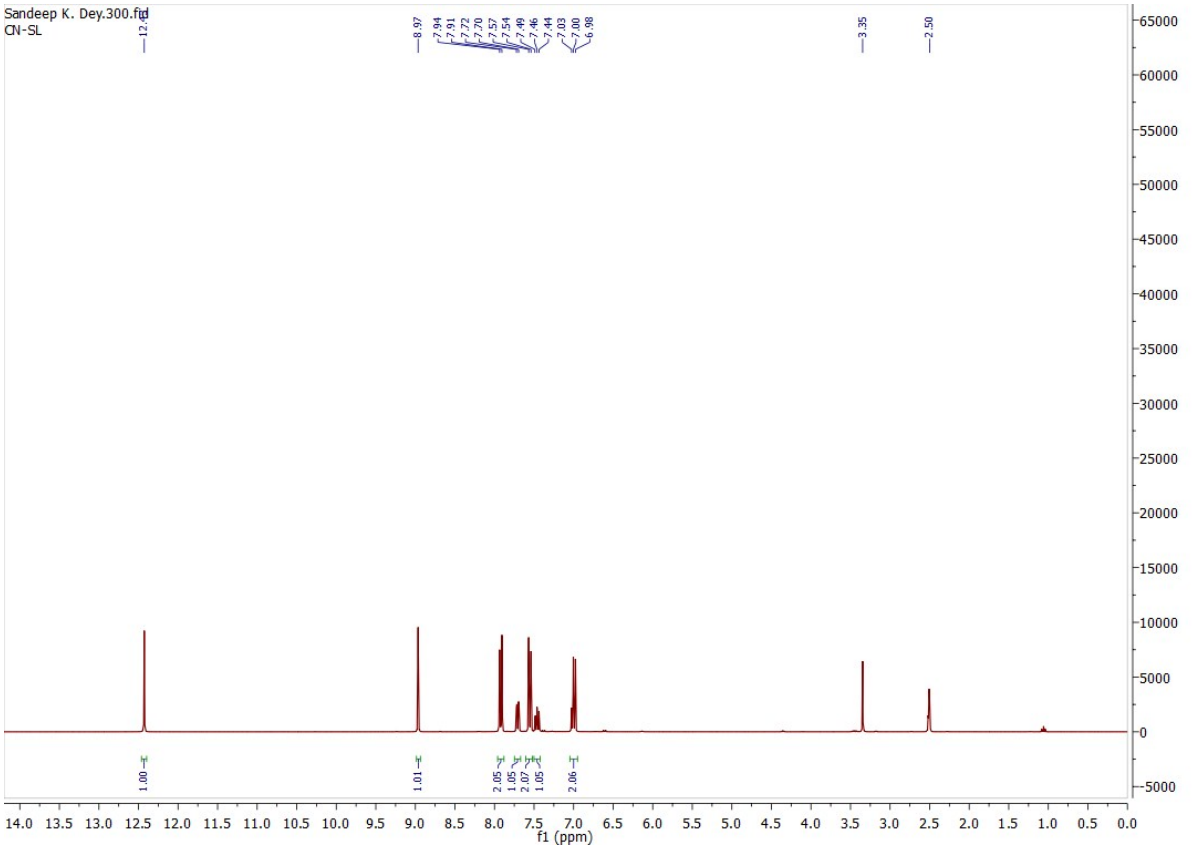

Fig. S8: H-NMR spectrum of **CL1** in DMSO-d_6.

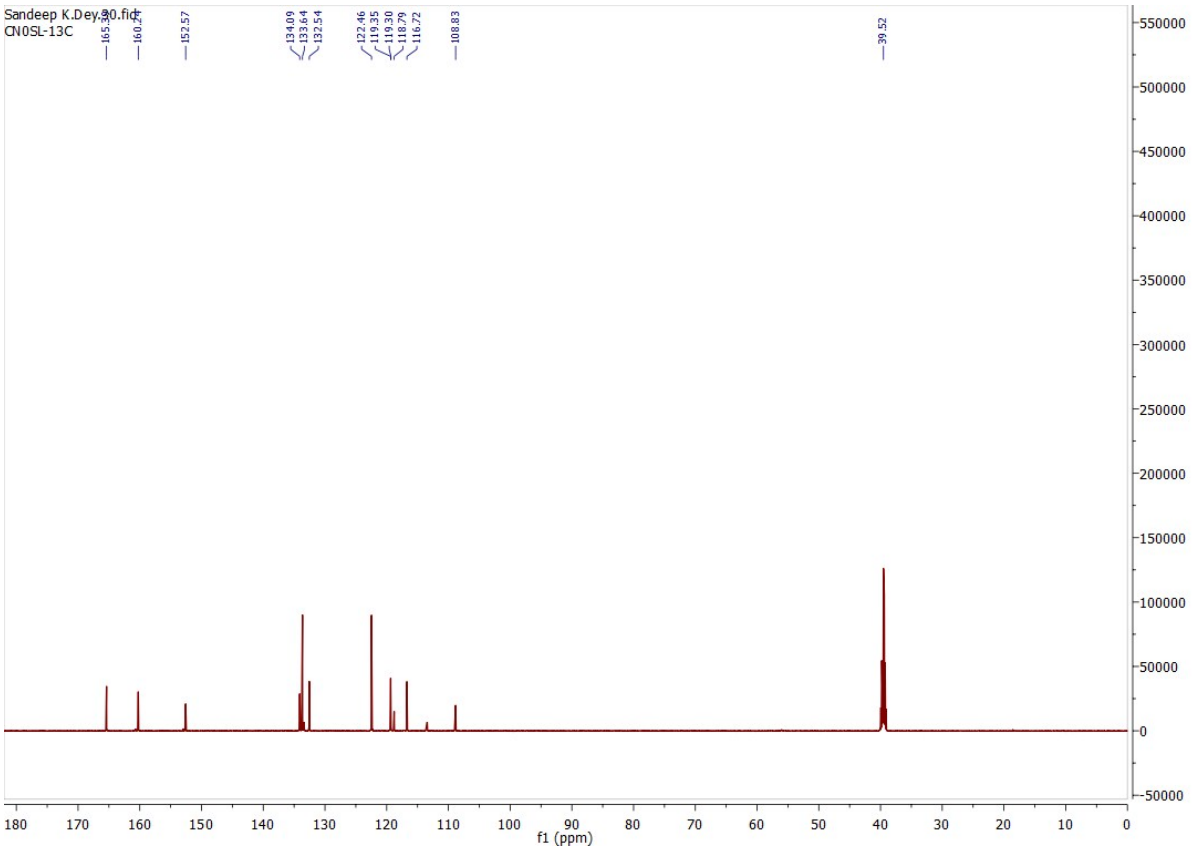

Fig. S9: ^{13}C-NMR spectrum of **CL1** in DMSO-d_6.

Mass Spectrum SmartFormula Report

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Sample Name	S. Dey CL-1 in Aceton (CH3OH)	Instrument	maXis 288882.20213
Comment			

Acquisition Parameter

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Scan End	1500 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Source

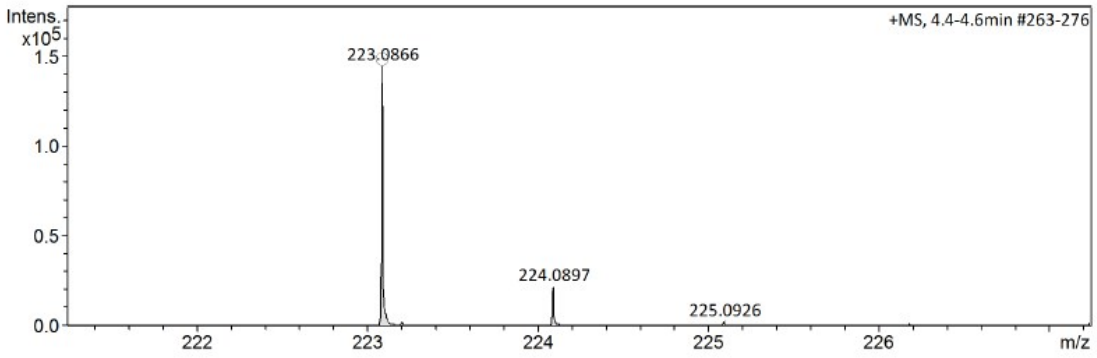

Fig. S10: ESI HR-MS of **CL1** in acetone. Peak at m/z 223.06 corresponds to (**CL1**+H^+).

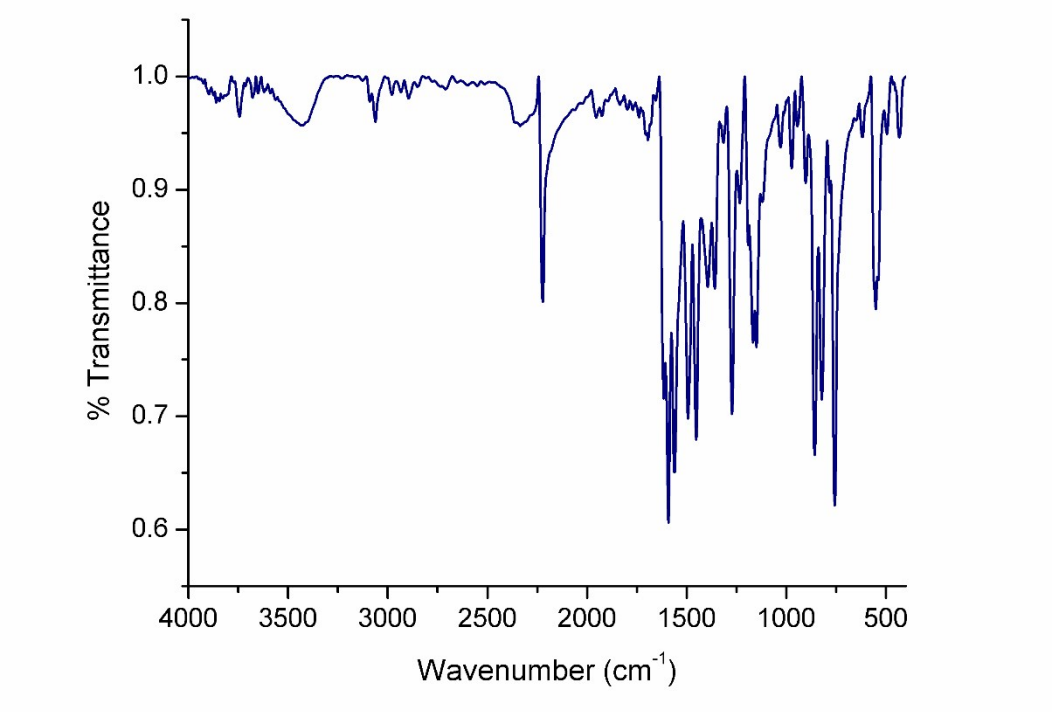

Fig. S11: FT-IR spectrum of **CL1** (KBr).

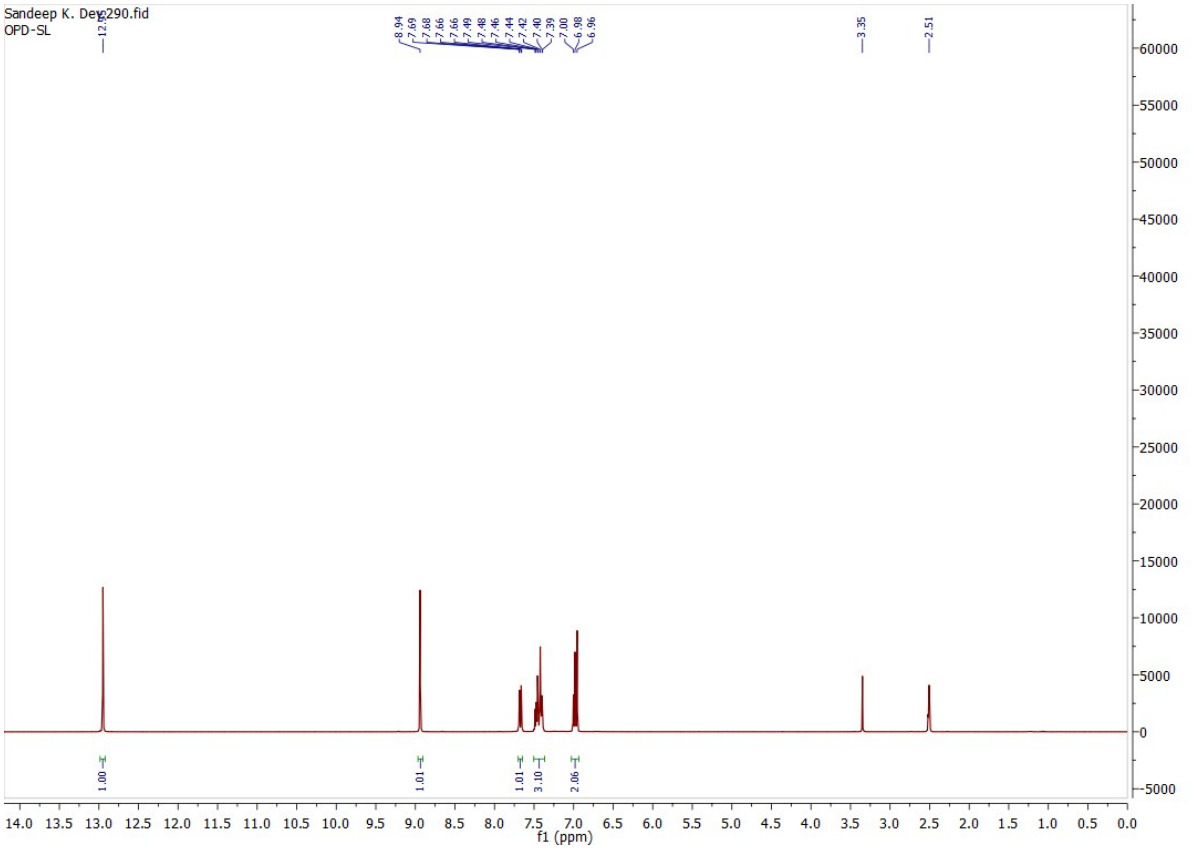

Fig. S12: ^{1}H-NMR spectrum of **CL2** in DMSO-d_6.

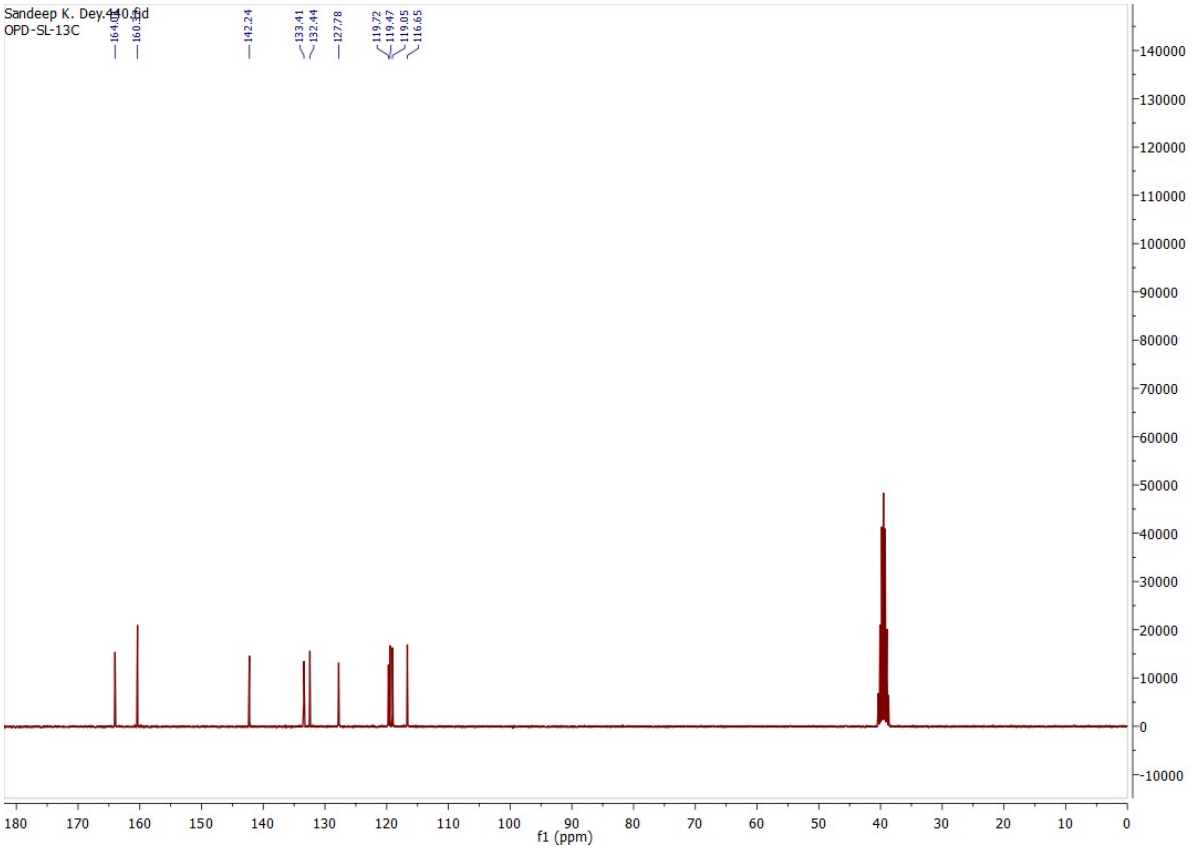

Fig. S13: ^{13}C-NMR spectrum of **CL2** in DMSO-d_6.

Mass Spectrum SmartFormula Report

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Sample Name	S. Dey CL-2 in Aceton (CH3OH)	Instrument	maXis 288882.20213
Comment			

Acquisition Parameter

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Scan End	1500 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Source

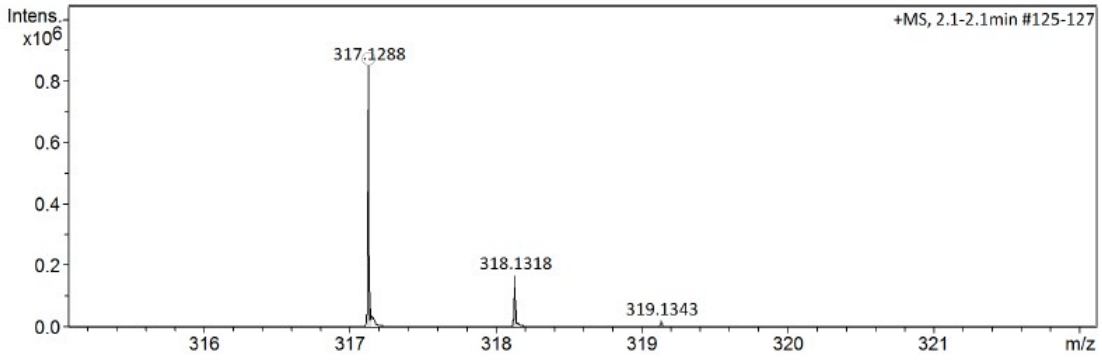

Fig. S14: ESI HR-MS of **CL2** in acetone. Peak at m/z 317.12 corresponds to (**CL2**+H^+).

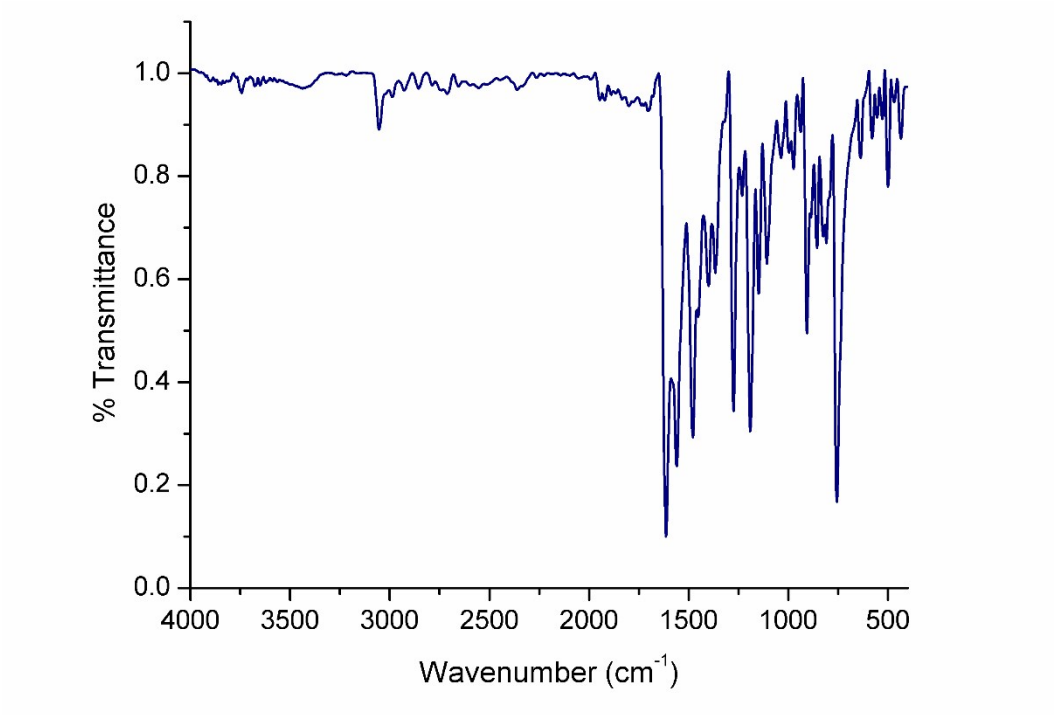

Fig. S15: FT-IR spectrum of **CL2** (KBr).

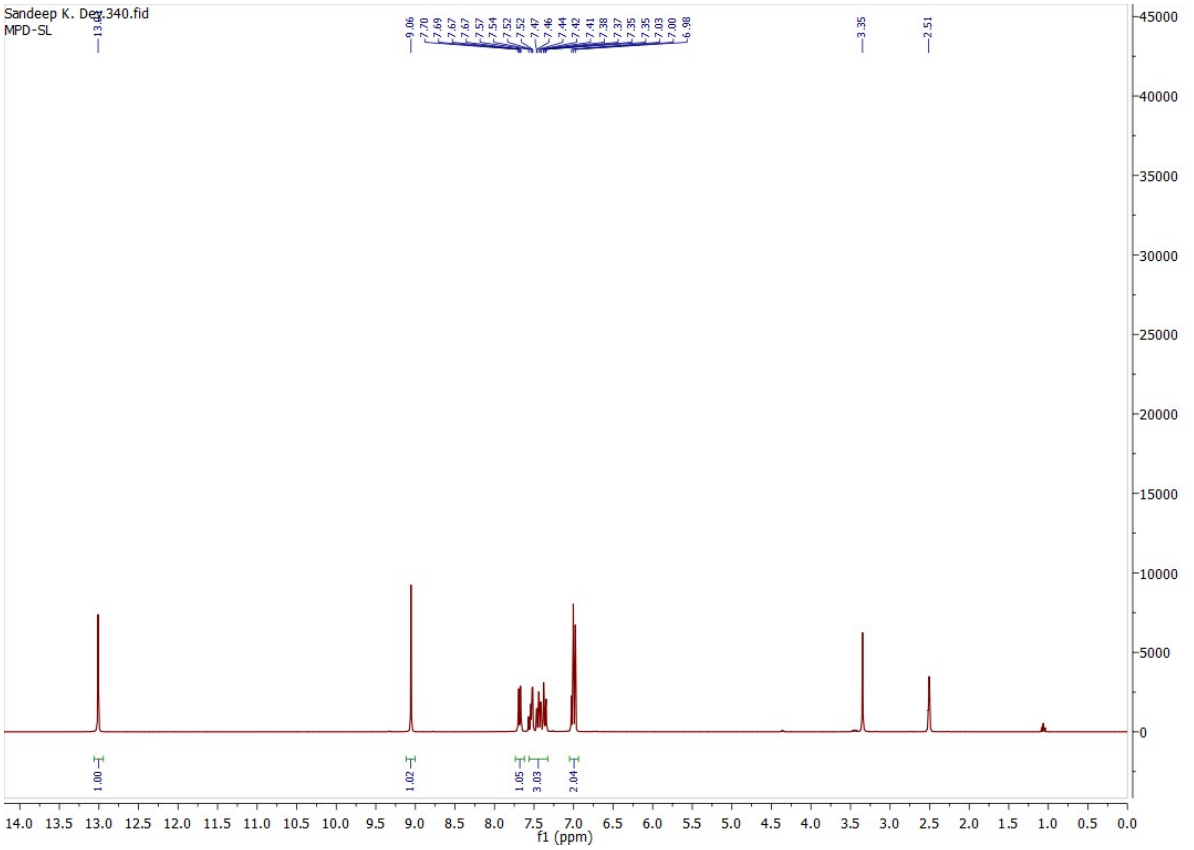

Fig. S16: ^{1}H-NMR spectrum of **CL3** in DMSO-d_6.

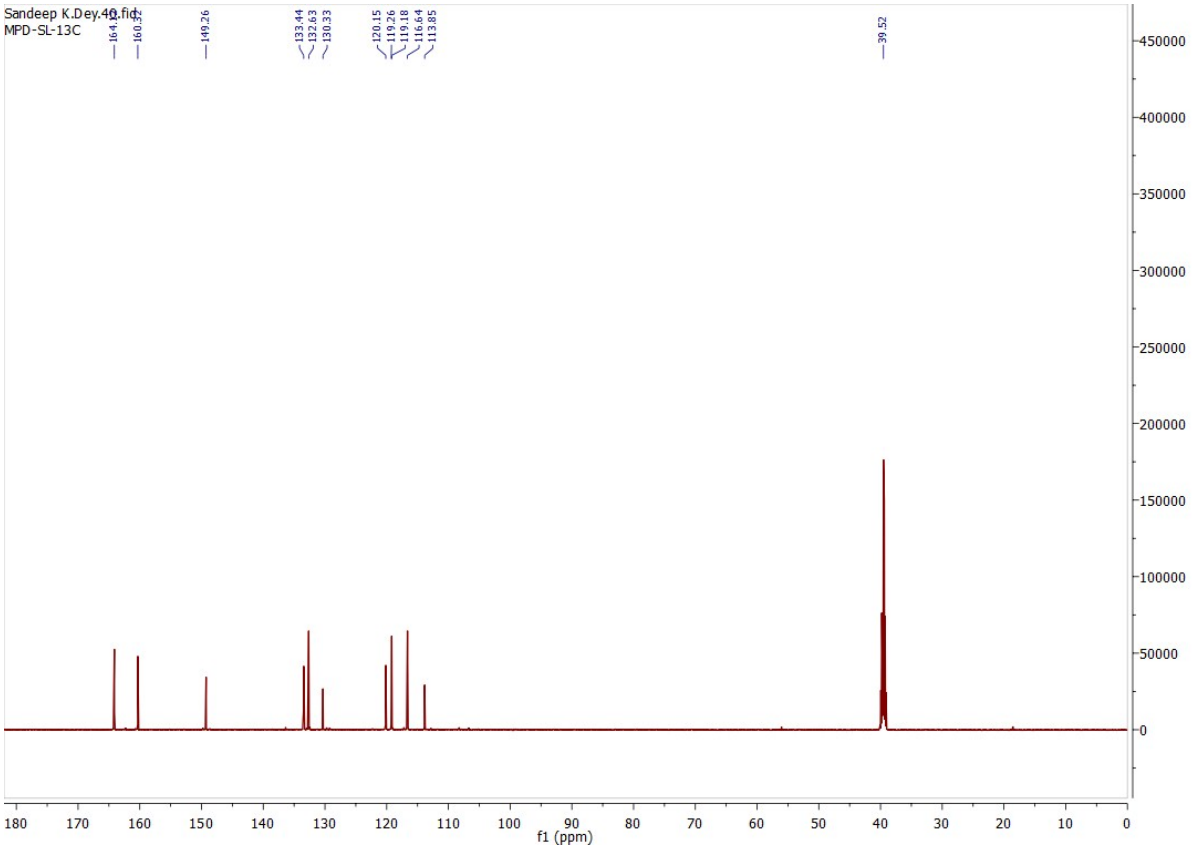

Fig. S17: ^{13}C-NMR spectrum of **CL3** in DMSO-d_6.

Mass Spectrum SmartFormula Report

Analysis Info

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Sample Name	S. Dey CL-3 in Aceton (CH3OH)	Instrument	maXis 288882.20213
Comment			

Acquisition Parameter

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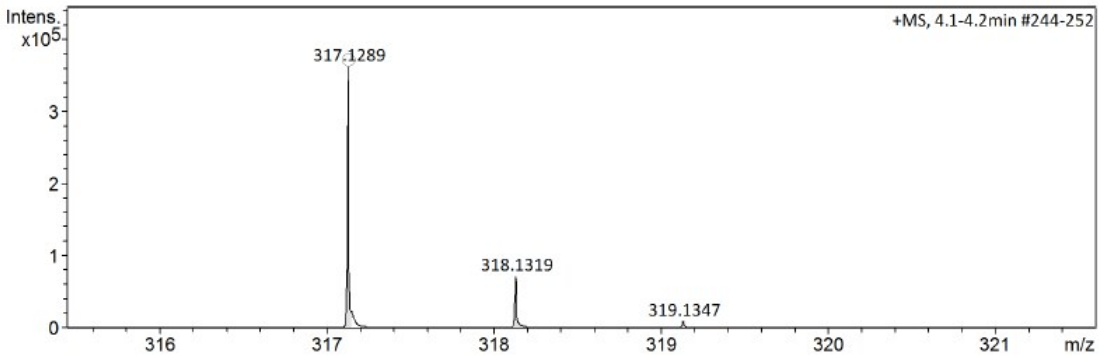


Fig. S18: ESI HR-MS of **CL3** in acetone. Peak at m/z 317.12 corresponds to (**CL3**+H^+).

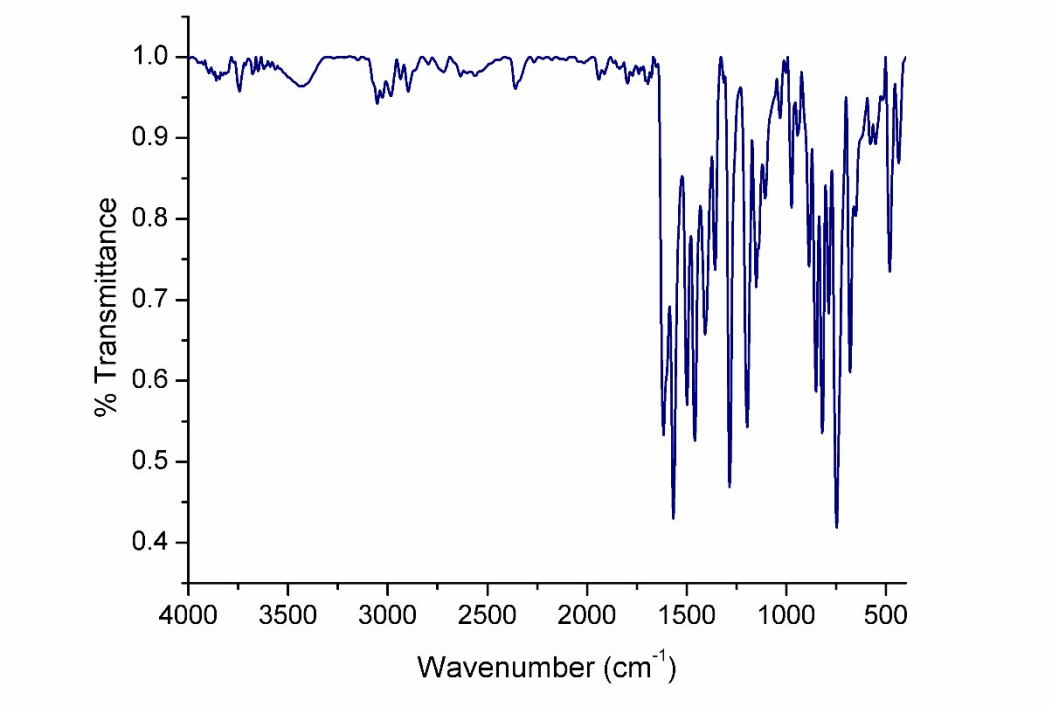


Fig. S19: FT-IR spectrum of **CL3** (KBr).

2. Experimental data (NMR and ESI-MS) for hydrolysis of SL in the presence of fluoride salts

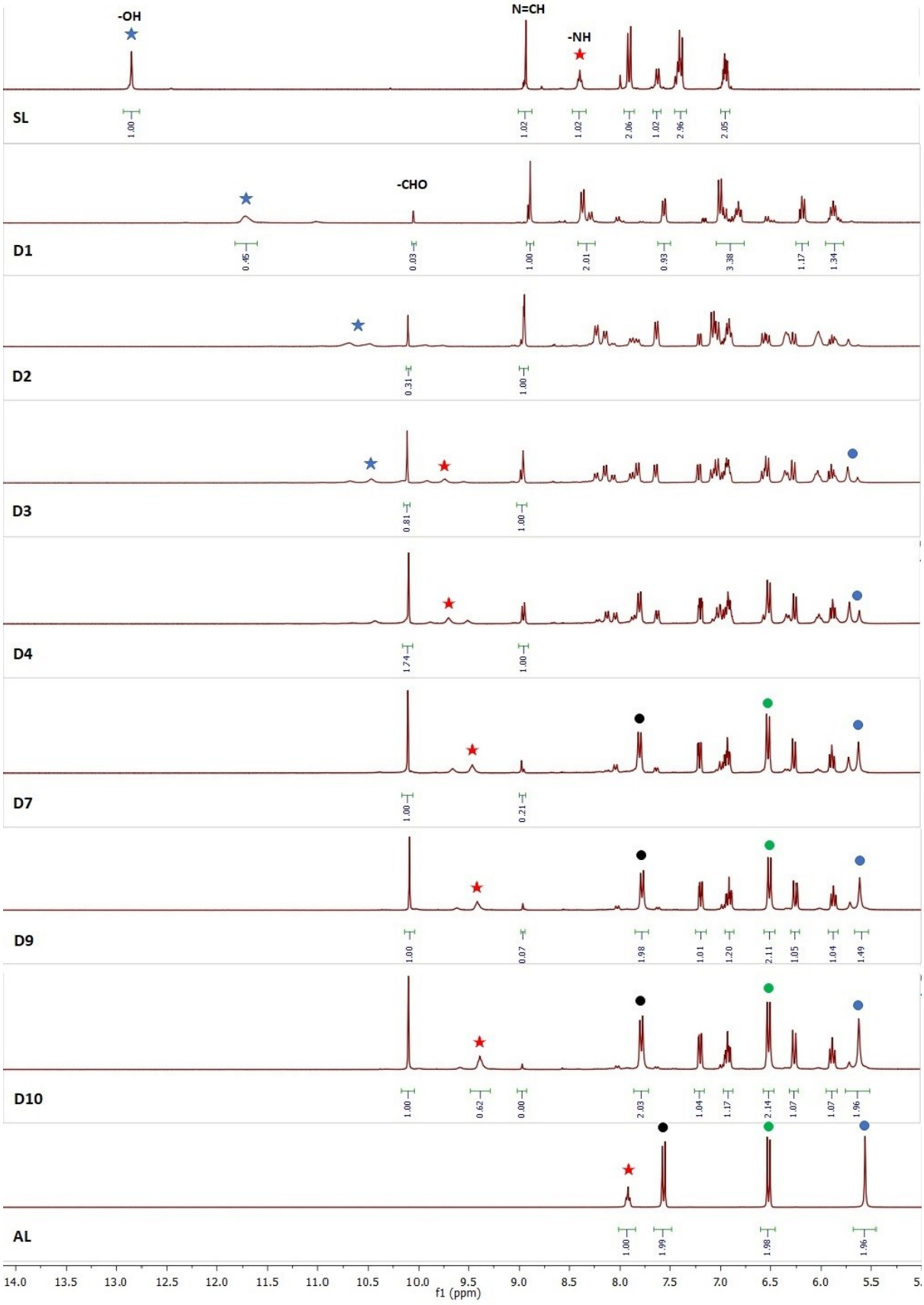


Fig. S20: ^{1}H-NMR spectra (DMSO-d_6) showing hydrolysis of **SL** in the presence of TBAF (10 equiv.) and compared with the ^{1}H-NMR spectrum of **AL**. D1, D2,……D10 indicates spectrum of **SL** mixed with TBAF recorded on day1 (within an hour), day2 (after 24 hrs.),……day10 respectively. Signal labelled with blue star represents -OH proton which is upfield shifted in presence of TBAF and gradually disappears on successive days due to deprotonation. Signal labelled with red star represents amide -NH proton which first disappear in the presence of TBAF and later reappears on successive days, however, downfield shifted. Signals labelled with blue, green and black circles represents -NH_2, *meta*-CH and *ortho*-CH (ortho and meta with respect to the amide group of **AL**) protons of **AL**. Signals which are not labelled in D10 spectrum are the aromatic protons of salicylaldehyde. Peak integral values of -CHO and -N=CH protons are given to calculate the percentage of hydrolysis of **SL**, discussed in the main text.

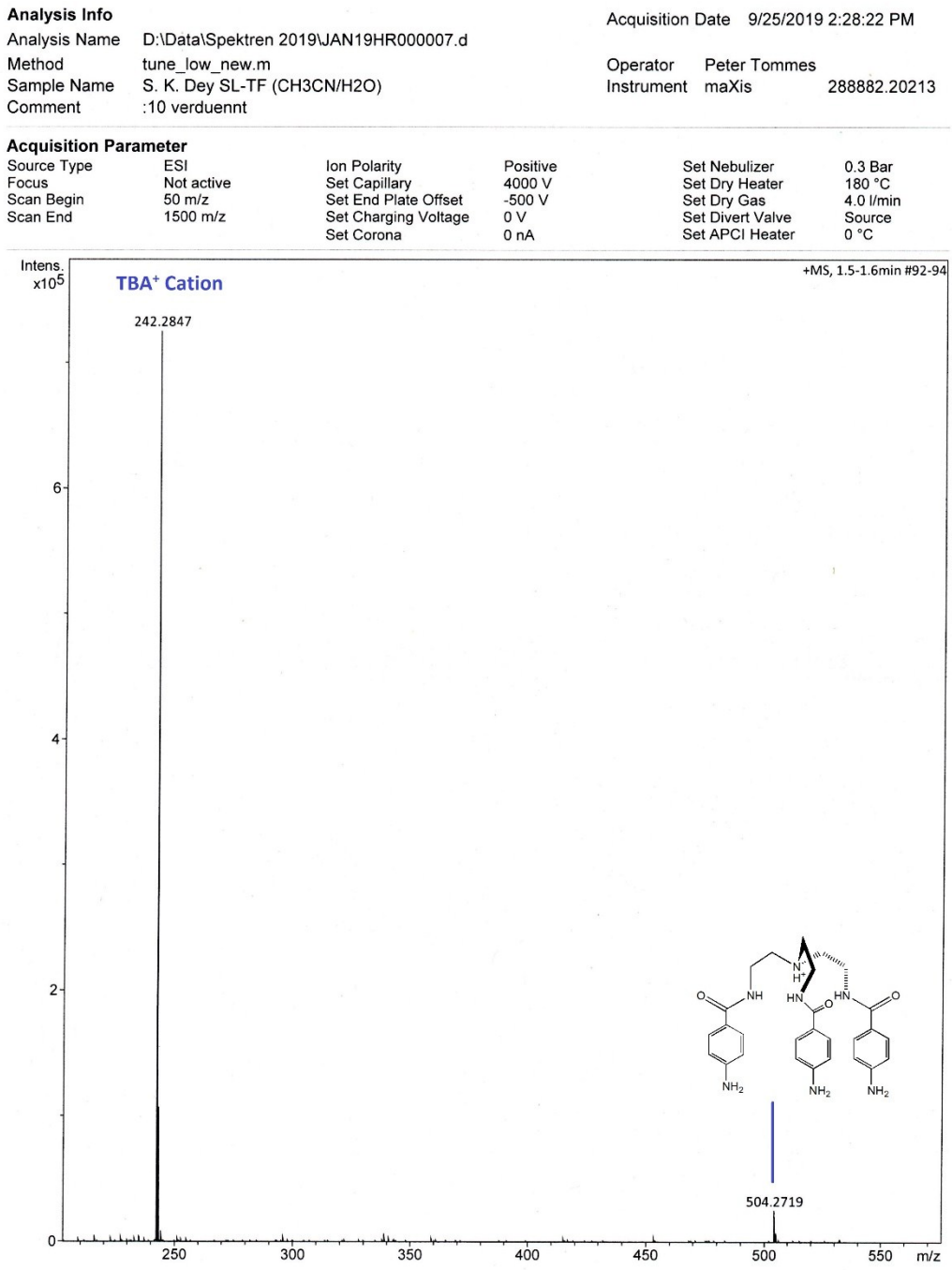


Fig. S21: LC-MS spectrum of **SL** in acetonitrile mixed with TBAF recorded after 10 days of mixing. Peak at m/z 504.27 corresponds to (**AL**+H^+).

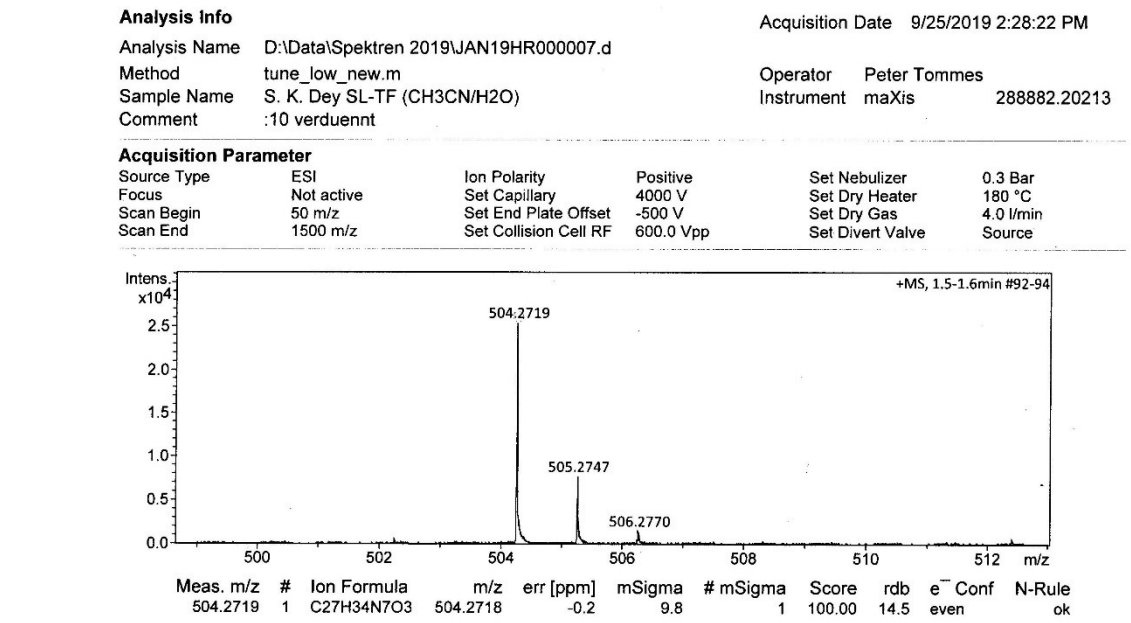
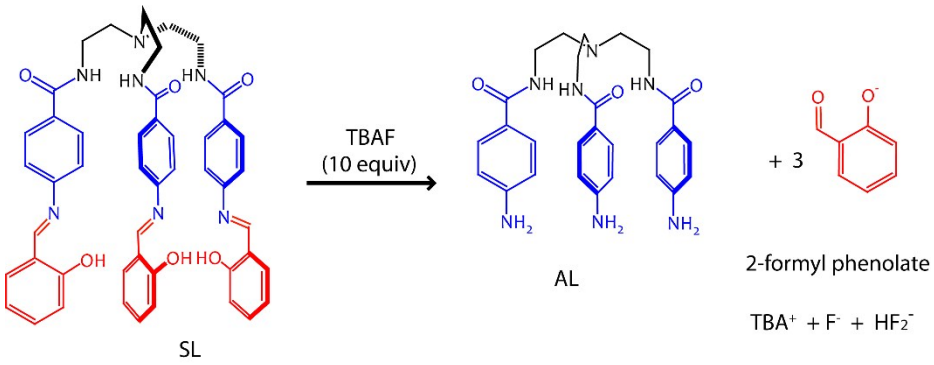


Fig. S22: LC-mass spectrum of **SL** in acetonitrile mixed with TBAF (expanded) recorded after 10 days of mixing. Peak at m/z 504.27, 505.27 and 506.27 corresponds to (**AL**+H^+),(**AL**+$2H^+$) and (**AL**+$3H^+$) respectively.



Scheme S2: Hydrolysis of **SL** to form 2-formyl phenolate (deprotonated salicylaldehyde) and **AL** as observed in ^{1}H-NMR and LC-MS experiments.

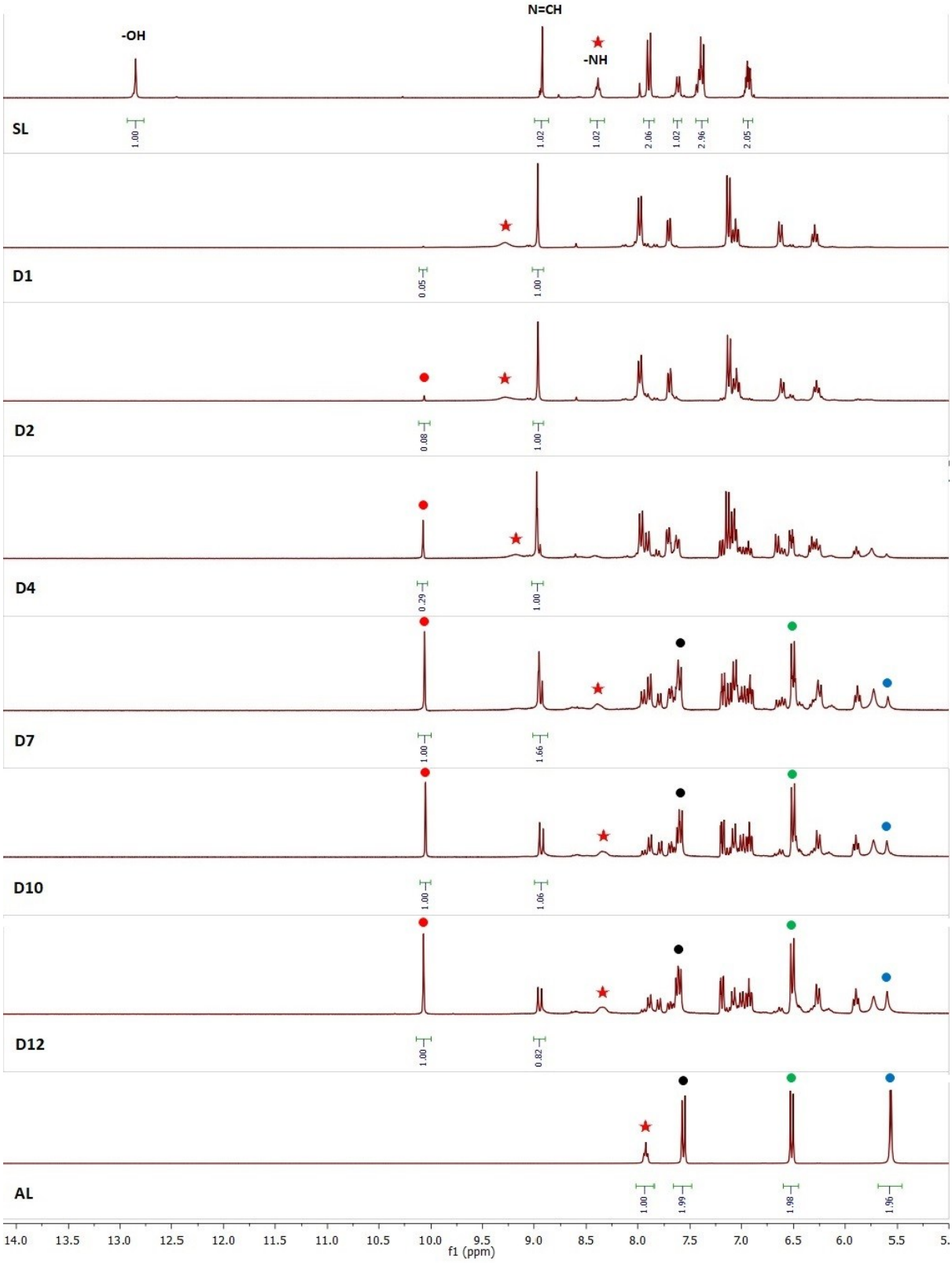


Fig. S23: ^{1}H-NMR spectra (DMSO-d_6) showing hydrolysis of **SL** in the presence of CsF (10 equiv.) and compared with the ^{1}H-NMR spectrum of **AL**. D1, D2,……D10 indicates spectrum of **SL** mixed with CsF recorded on day1 (within 1 hr.), day2 (after 24 hrs.),……day10 respectively. Signal labelled with red star represents amide -NH

proton which is initially downfield shifted in the presence of CsF and later experiences upfield shift on successive days, as hydrolysis of **SL** progress. Signals labelled with blue, green and black circles represents -NH_2, *meta*-CH and *ortho*-CH (ortho and meta with respect to the amide group of **AL**) protons of **AL**. Signals which are not labelled in the spectra are the aromatic protons of salicylaldehyde (hydrolysis product) and **SL**. Peak integral values of -CHO and N=CH protons are given to calculate the percentage of hydrolysis of **SL**, as discussed in the main text.

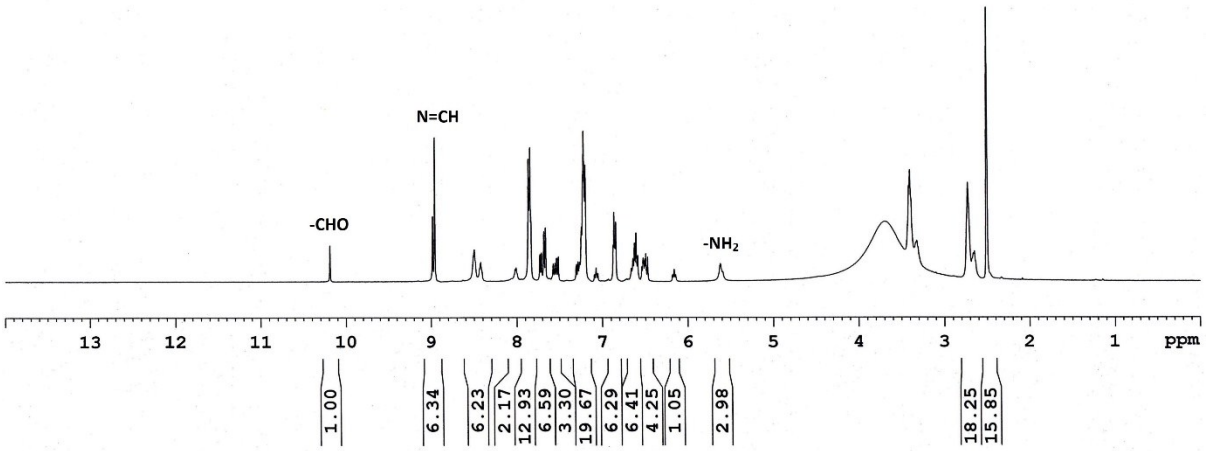


Fig. S24: ^{1}H-NMR spectrum (DMSO-d_6) showing hydrolysis of **SL** in the presence of KF.

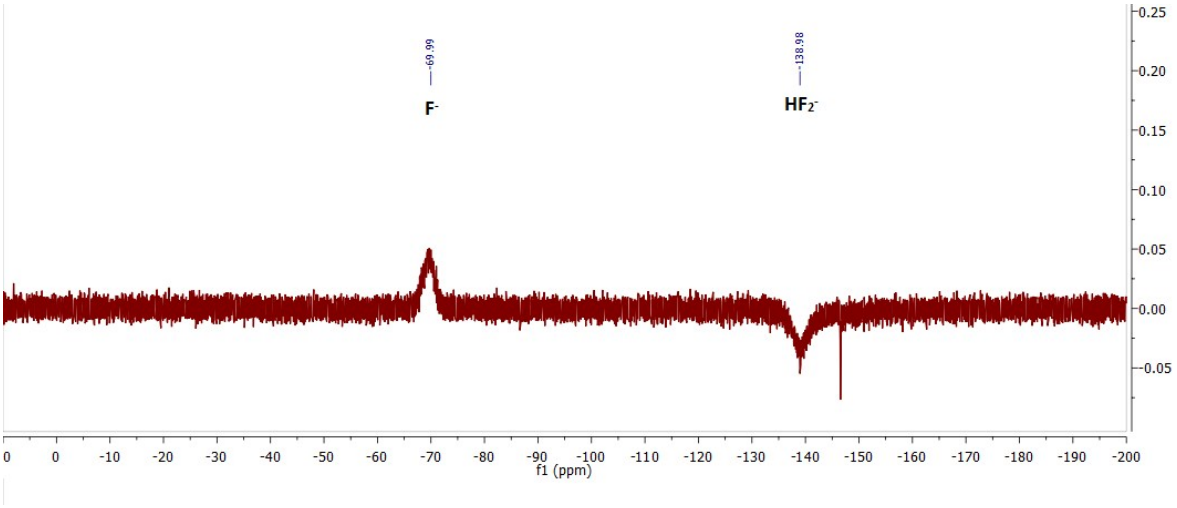


Fig. S25: ^{19}F-NMR spectrum of **SL** mixed with TBAF in DMSO-d_6, recorded after 10 days of mixing.

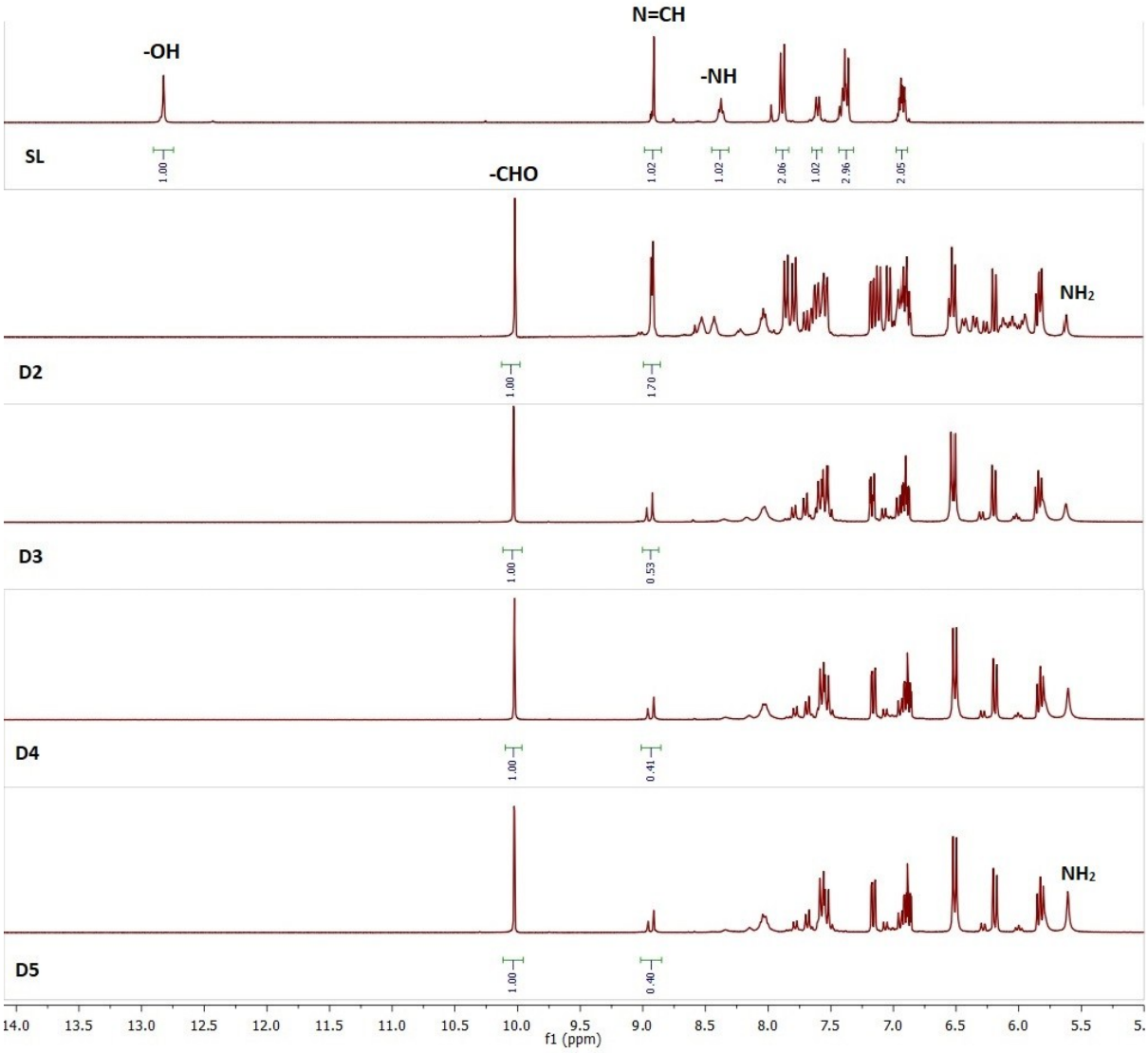


Fig. S26: ^{1}H-NMR spectra (DMSO-d_6) showing hydrolysis of **SL** in the presence of Cs_2CO_3 (3 equiv.). D2,D3,……D5 indicates spectrum of **SL** mixed with Cs_2CO_3 recorded on day2 (after 24 hrs.),day3 (after 48 hrs.)……day5 respectively. Salicylidene -OH proton signal disppeared upon addition of Cs_2CO_3. 70% hydrolysis was observed to be completed in 5 days with 3 equiv. of Cs_2CO_3.

3. Experimental data (NMR and ESI-MS) for hydrolysis of CL1 in the presence of fluoride salts

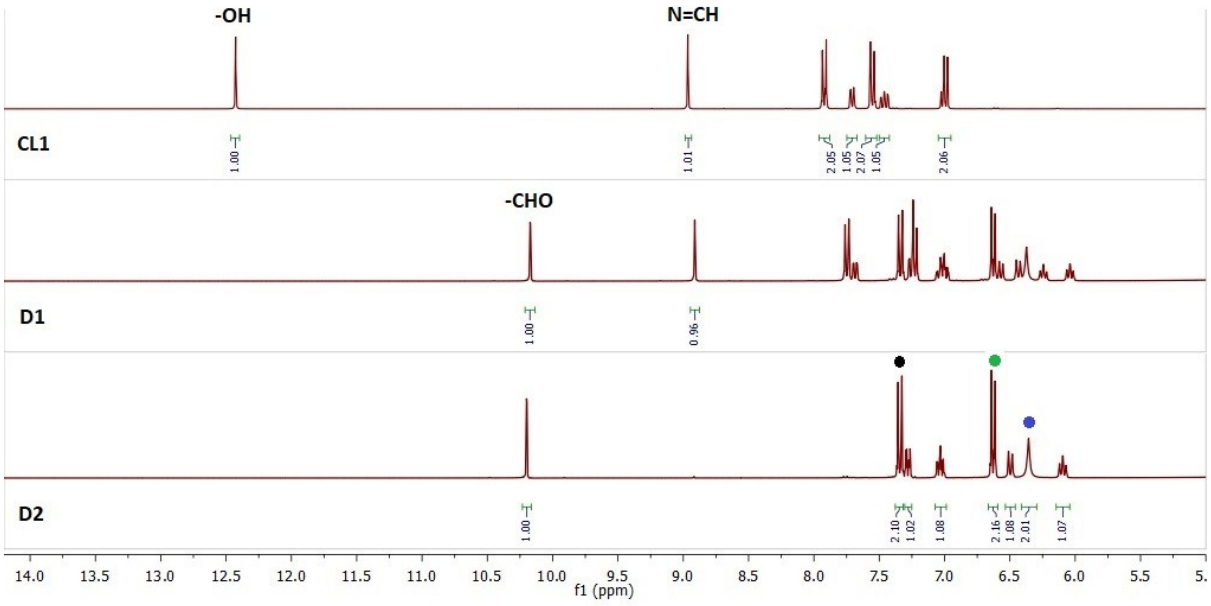


Fig. S27: ^{1}H-NMR spectra (DMSO-d_6) showing hydrolysis of **CL1** in the presence of TBAF (2 equiv.). D1, D2 indicates spectrum of **CL1** mixed with TBAF recorded on day1 (within 1 hr.), day2 (after 24 hrs.) respectively. Signals labelled with blue, green and black circles represents -NH_2, *ortho*-CH and *meta*-CH (ortho and meta with respect to -NH_2 group) protons of **2-aminobenzonitrile** (hydrolysis product). Signals which are not labelled in the spectrum D2 are the aromatic protons of salicylaldehyde (hydrolysis product). Peak integral values of -CHO and N=CH protons are given to calculate the percentage of hydrolysis of **CL1**, as discussed in the main text. 100% hydrolysis was observed to be completed in 24 hours (D2).

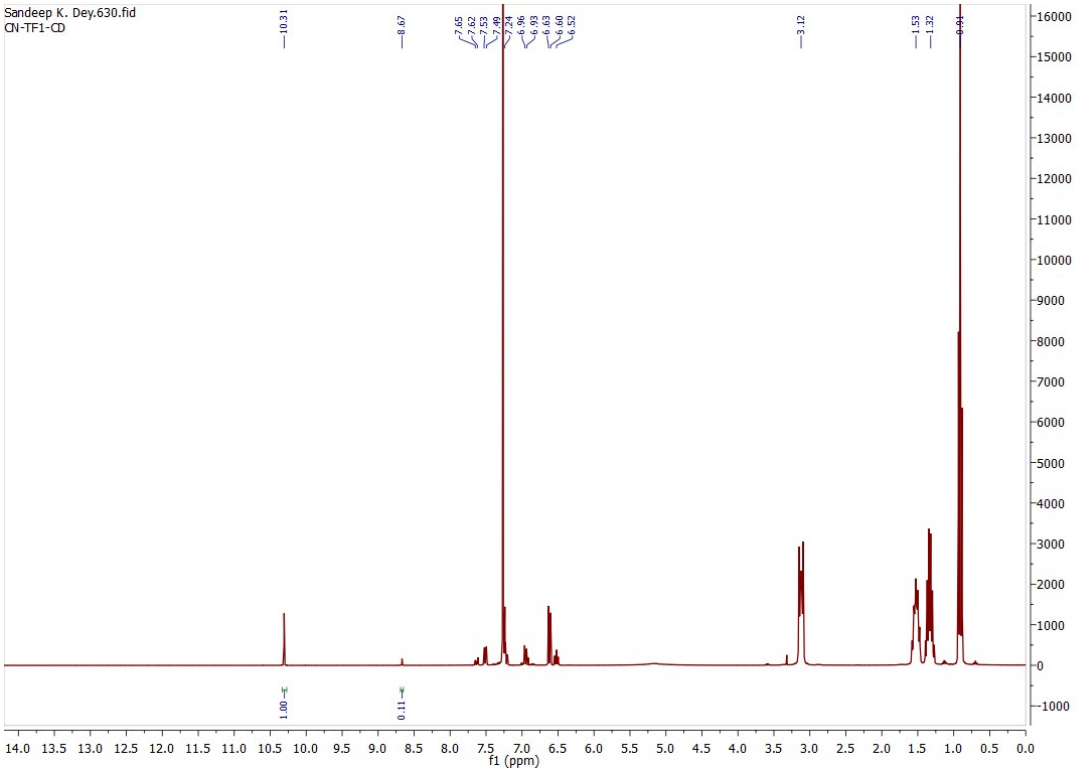
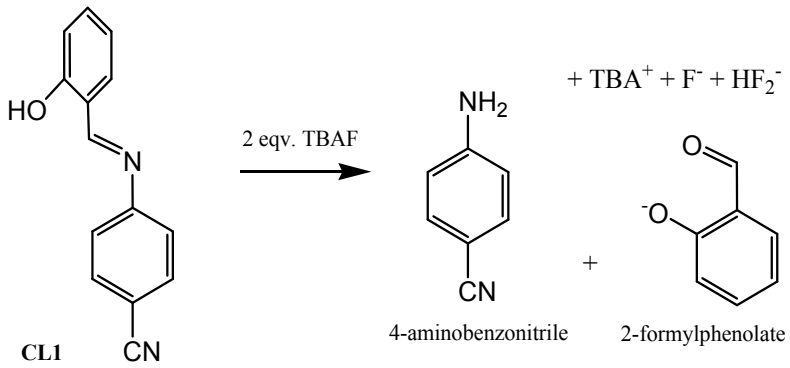


Fig. S28: ^{1}H-NMR spectrum ($CDCl_3$) showing hydrolysis of **CL1** in the presence of TBAF (2 equiv.). 90% hydrolysis was observed to be completed in 1 hour.



Scheme S3: Hydrolysis of **CL1** to form 2-formyl phenolate and 4-aminobenzonitrile as observed in NMR and LC-MS experiments.

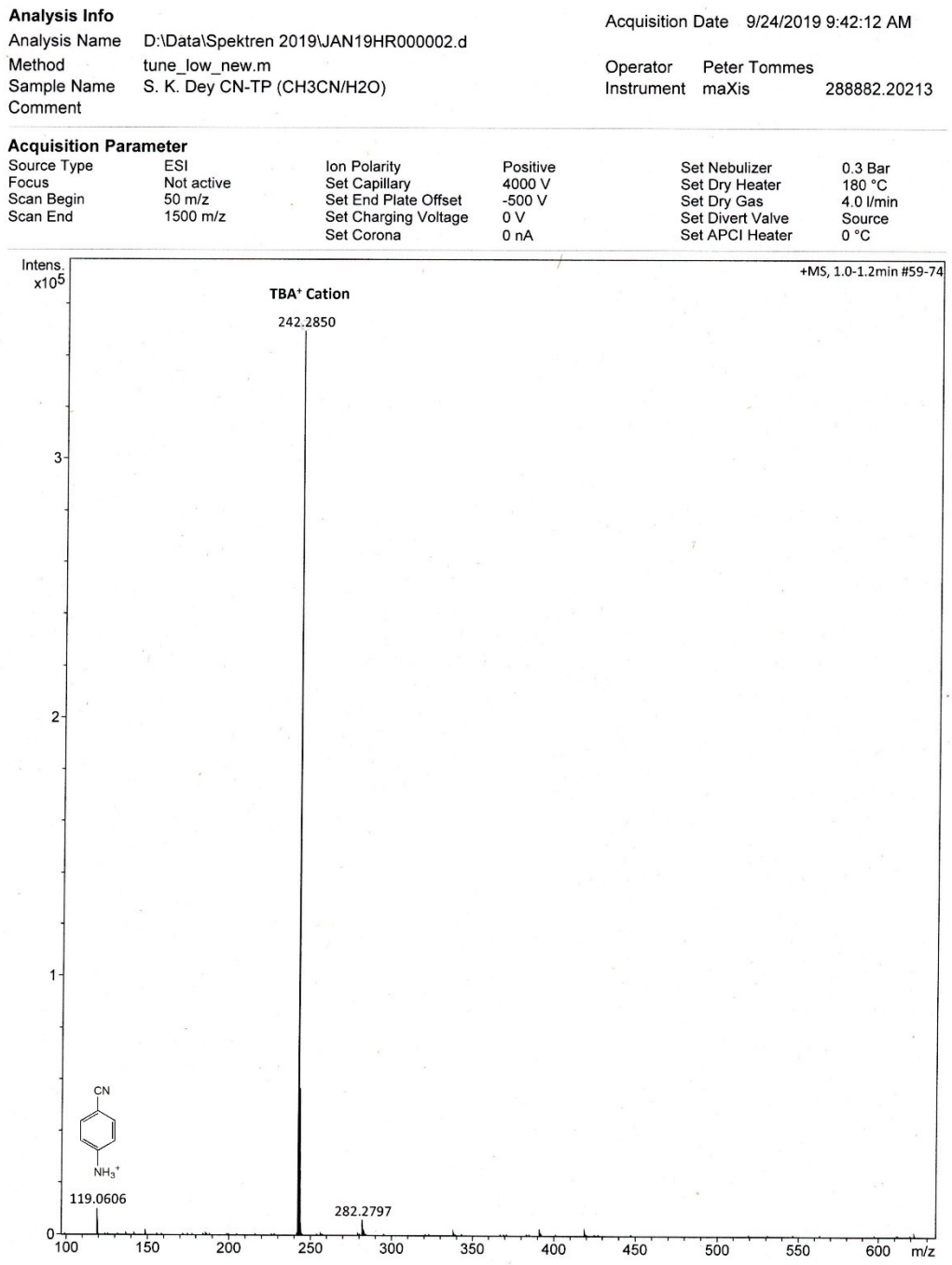


Fig. S29: LC-MS spectrum of **CL1** in acetonitrile mixed with TBAF recorded after 2 days of mixing. Peak at m/z 119.06 corresponds to (**CL1**+H^+).

4. Experimental data (NMR and ESI-MS) for hydrolysis of CL2 in the presence of fluoride salts

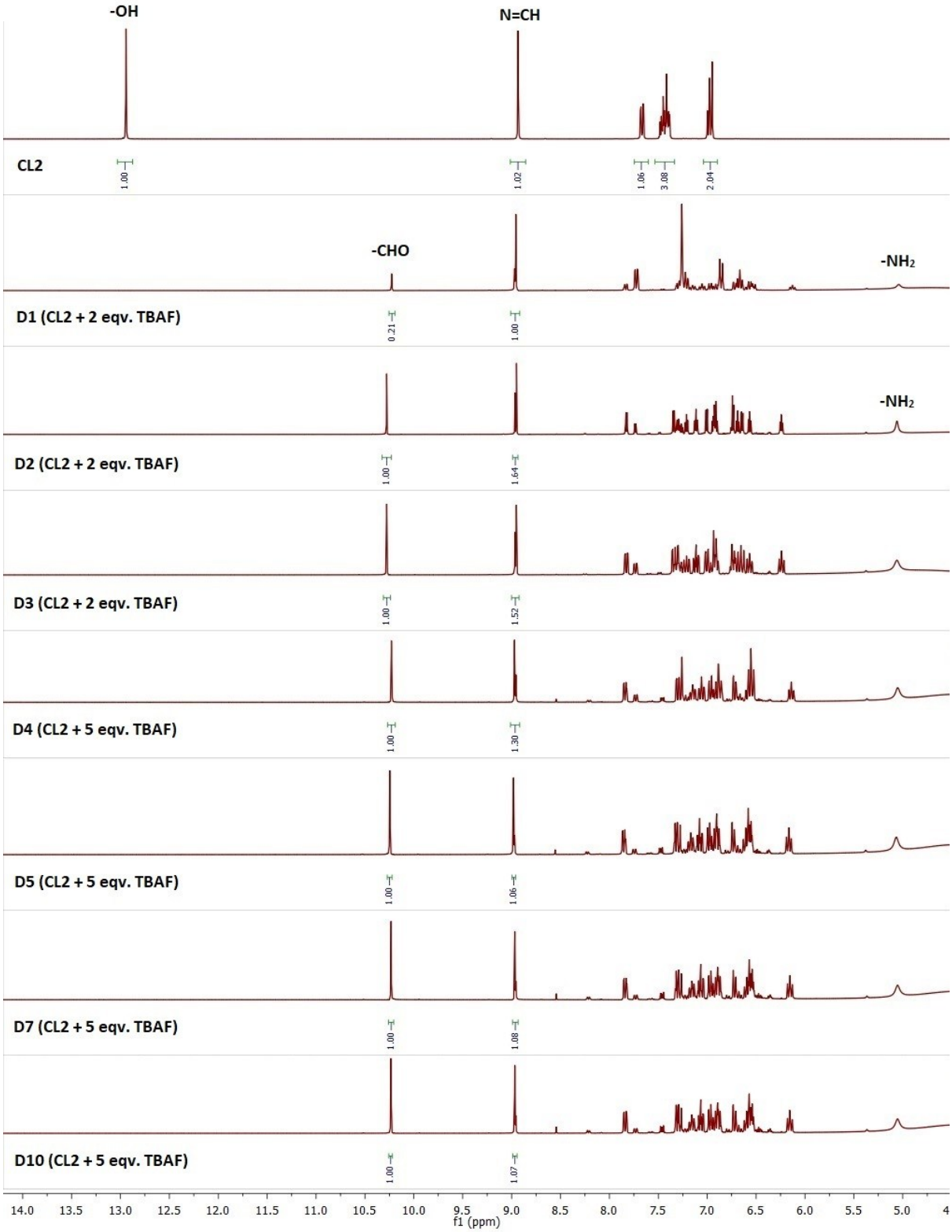


Fig. S30: ^{1}H-NMR spectra (DMSO-d_6) showing hydrolysis of **CL2** in the presence of TBAF (2 and 5 equiv.). D1, D2,…..D10 indicates spectrum of **CL2** mixed with TBAF recorded on day1(within 1 hr.), day2 (after 24 hrs.),…..day10 respectively. Peak integral values of -CHO and N=CH protons are given to calculate the percentage of hydrolysis of **CL2**, as discussed in the main text. No more than 50% hydrolysis was observed in presence of excess TBAF.

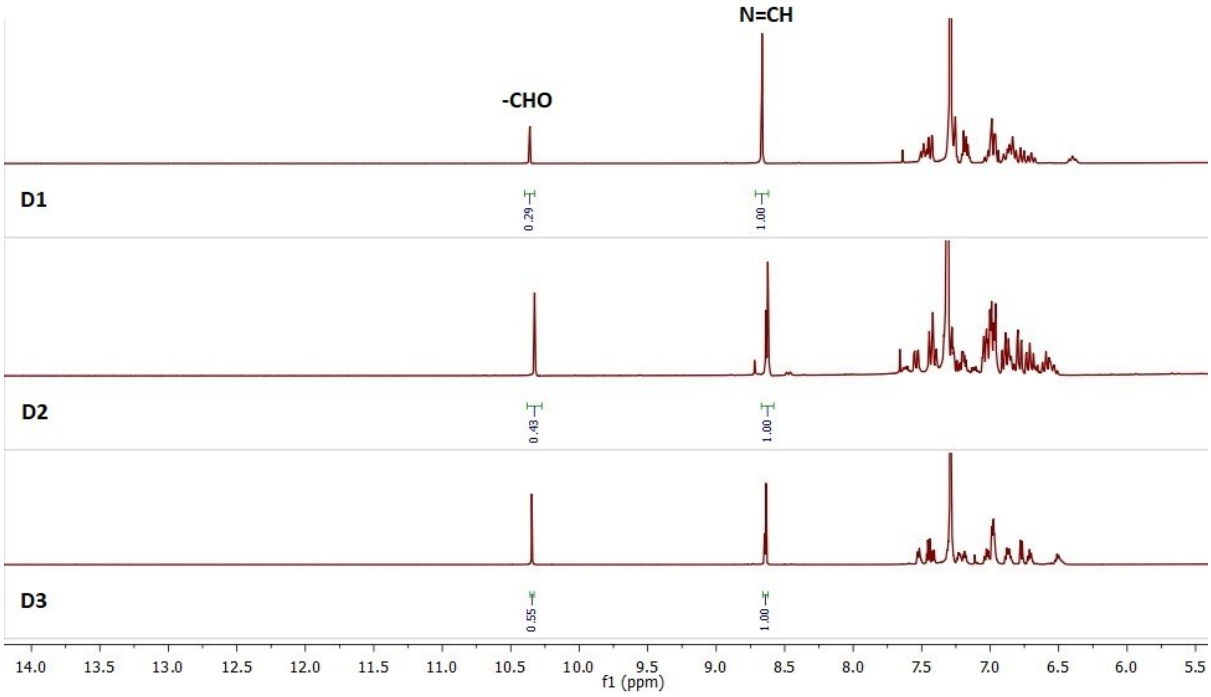


Fig. S31: ^{1}H-NMR spectra ($CDCl_3$) showing hydrolysis of **CL2** in the presence of TBAF (2 equiv.). D1, D2, D3 indicates spectrum of **CL2** mixed with TBAF recorded on day1, day2, day3 respectively. Peak integral values of -CHO and N=CH protons are given to calculate the percentage of hydrolysis of **CL2.**

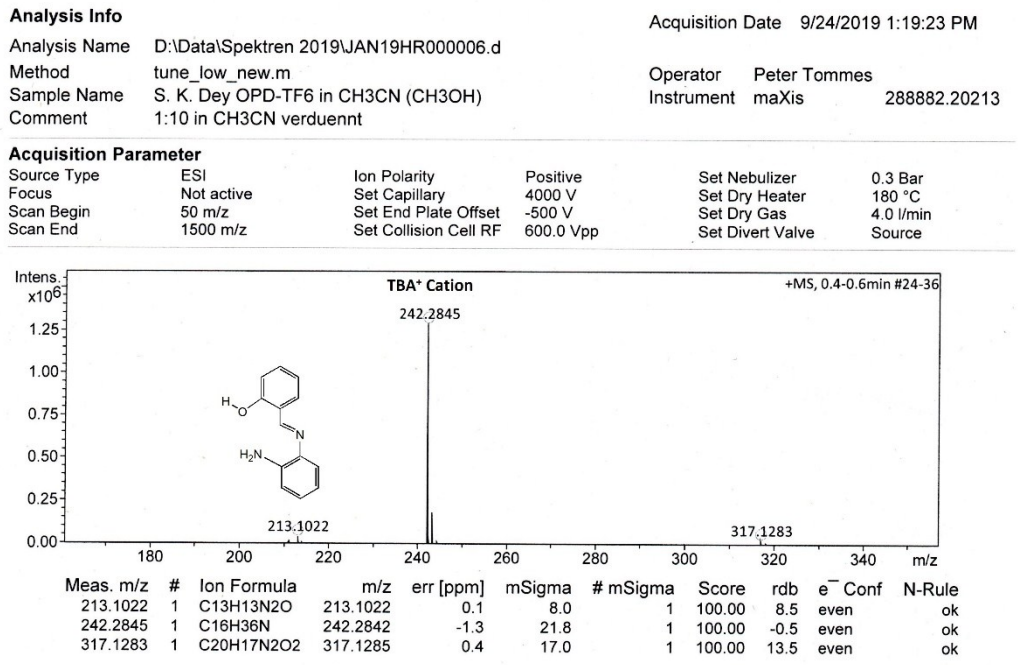


Fig. S32: LC-MS spectrum of **CL2** in acetonitrile mixed with TBAF recorded after 6 days of mixing.

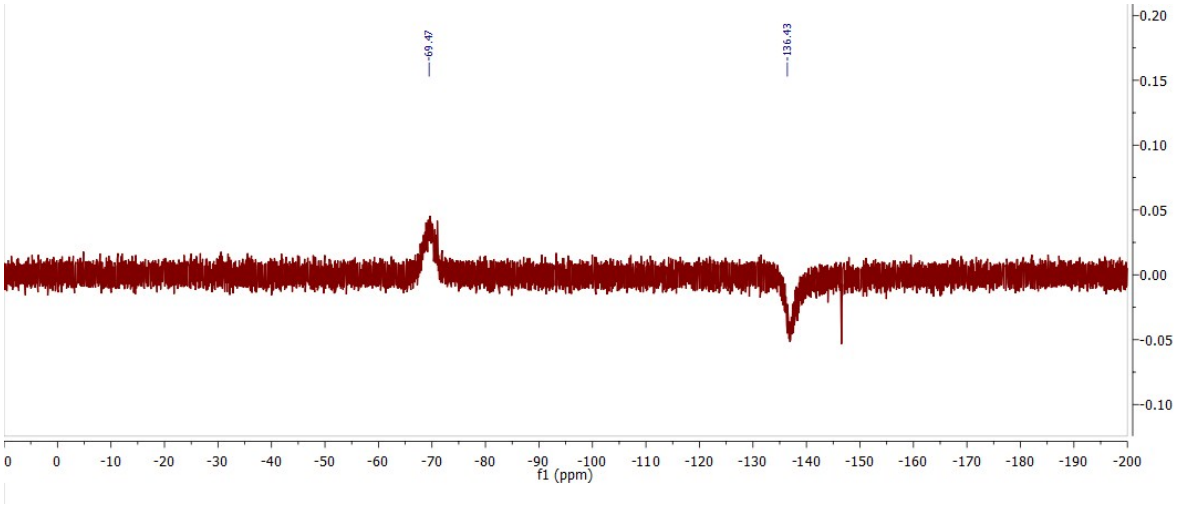
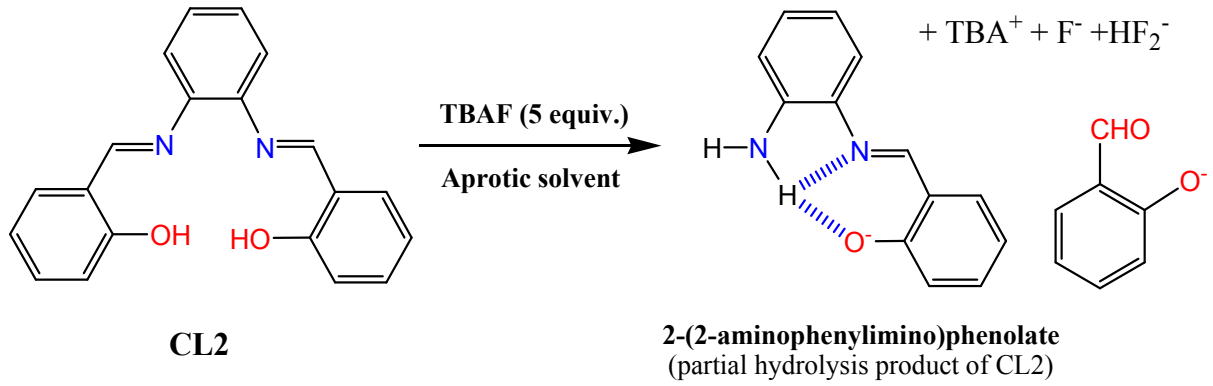


Fig. S33: ^{19}F-NMR spectrum of **CL2** mixed with TBAF in DMSO-d_6, recorded after 10 days of mixing.



Scheme S4: Partial hydrolysis of **CL2** to form 2-formyl phenolate and 2(2-aminophenylimino)phenolate as observed in ^{1}H-NMR and LC-MS experiments. Formation of intramolecular H-bond between -NH_2 and phenolate oxygen in 2(2-aminophenylimino)phenolate might resist the hydrolysis of the second imine bond in **CL2**. HF_2^- anion is formed in situ in the solution mixture of **CL2** and TBAF.

5. Experimental data (NMR and LC-MS) for hydrolysis of CL3 in the presence of fluoride salts

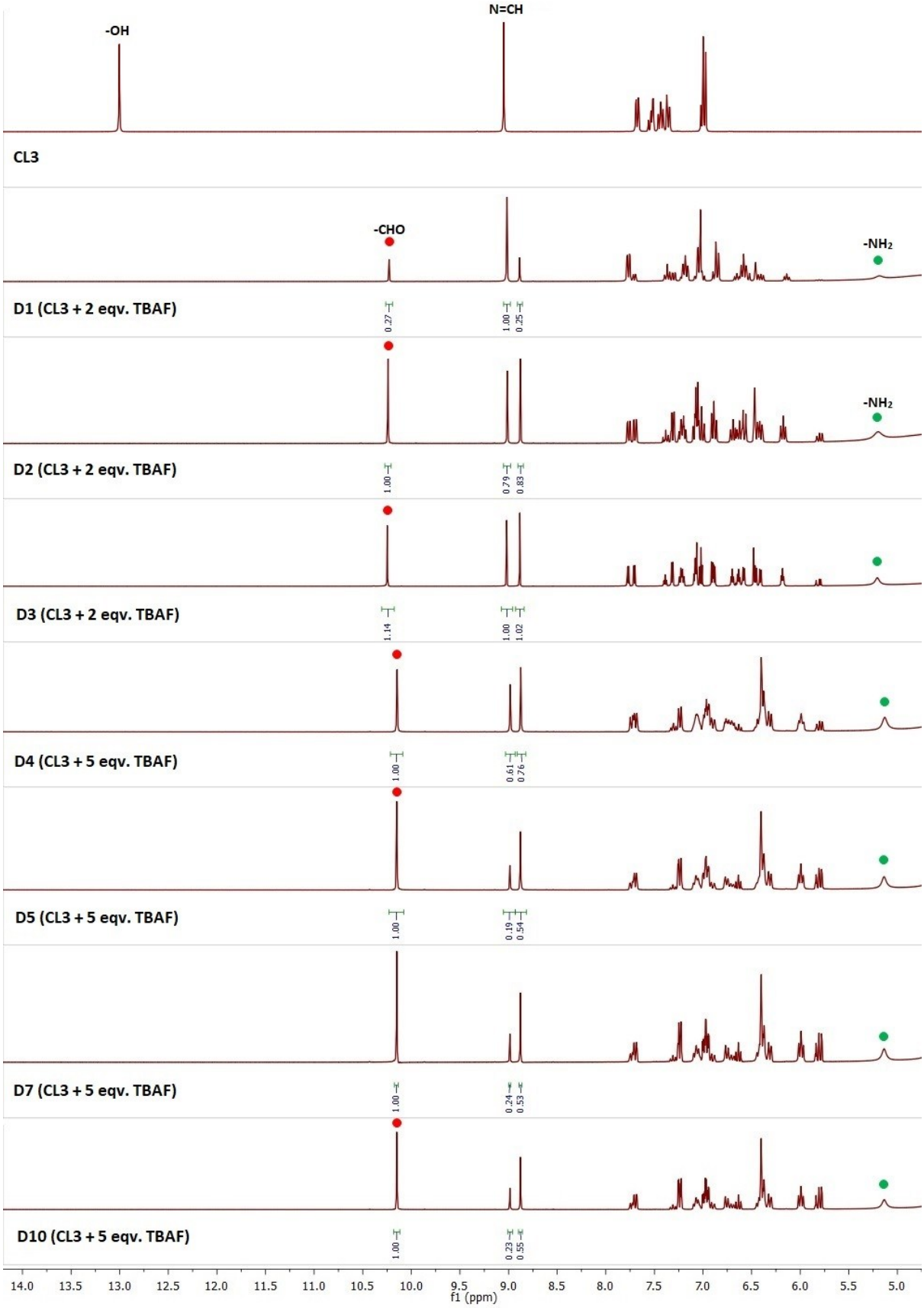


Fig. S34: ^{1}H-NMR spectra (DMSO-d_6) showing hydrolysis of **CL3** in the presence of TBAF (2 and 5 equiv.). D1, D2,…..D10 indicates spectrum of **CL3** mixed with TBAF recorded on day1 within 1 hr.), day2 (after 24 hrs.),…..day10 respectively. Peak integral values of -CHO and N=CH protons are given to calculate the percentage of hydrolysis of **CL3**.

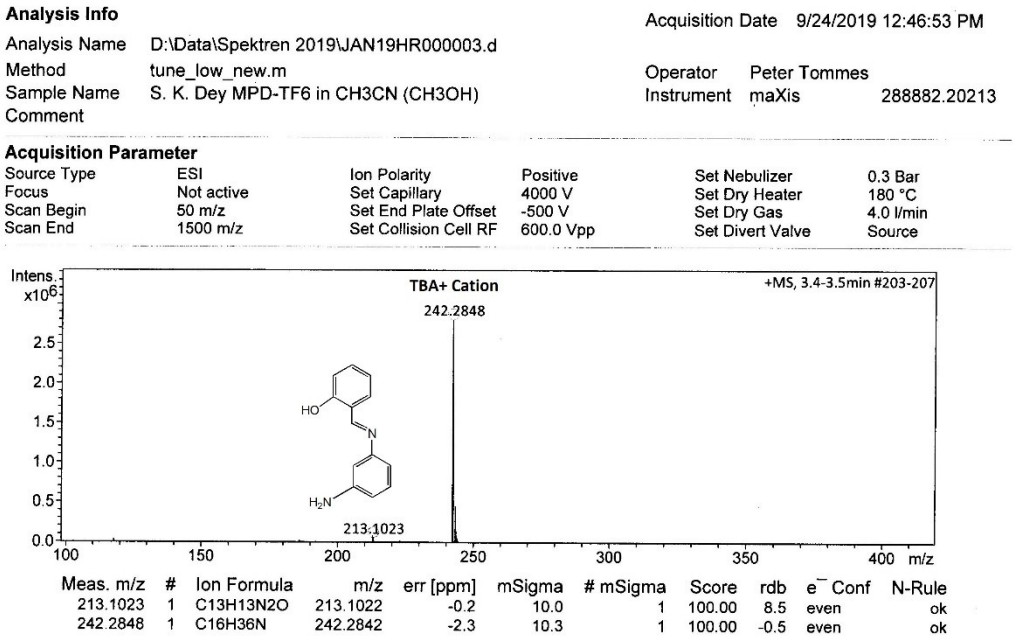
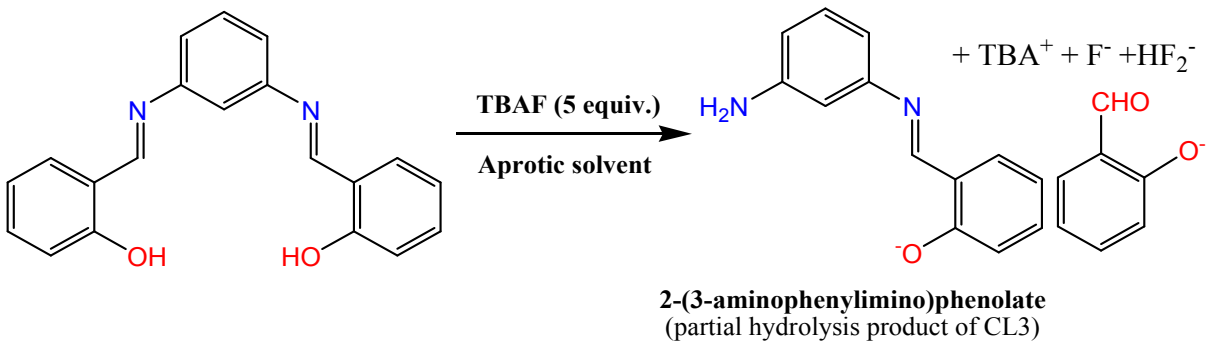


Fig. S35: LC-MS spectrum of **CL3** in acetonitrile mixed with TBAF recorded after 6 days of mixing.



Scheme S5: Partial hydrolysis of **CL3** to form 2-formyl phenolate and 2(3-aminophenylimino)phenolate as observed in H-NMR and LC-MS experiments.

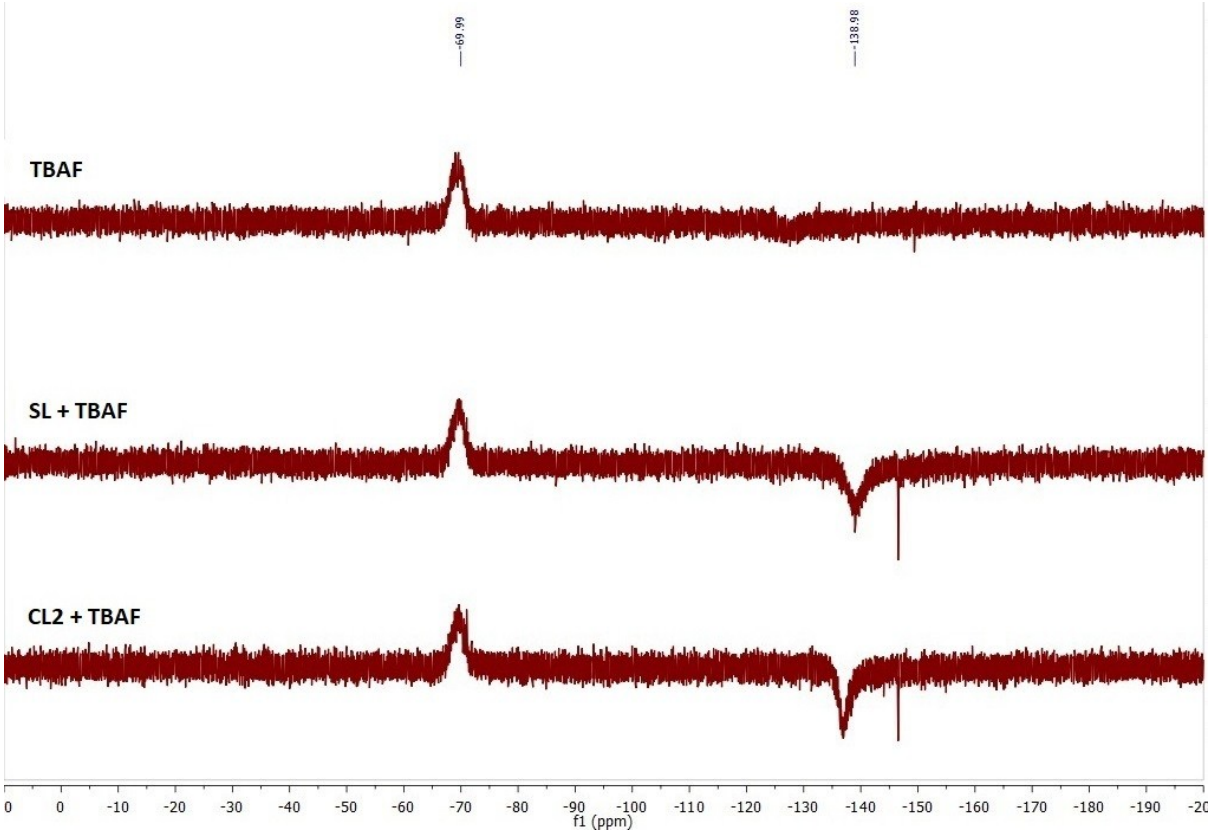


Fig. S36: ^{19}F NMR (DMSO-d_6) of TBAF, **SL** mixed with TBAF, and **CL2** mixed with TBAF. The peak at -69 corresponds to free fluoride anion and the peak at -138 corresponds to hydrogen difluoride anion.

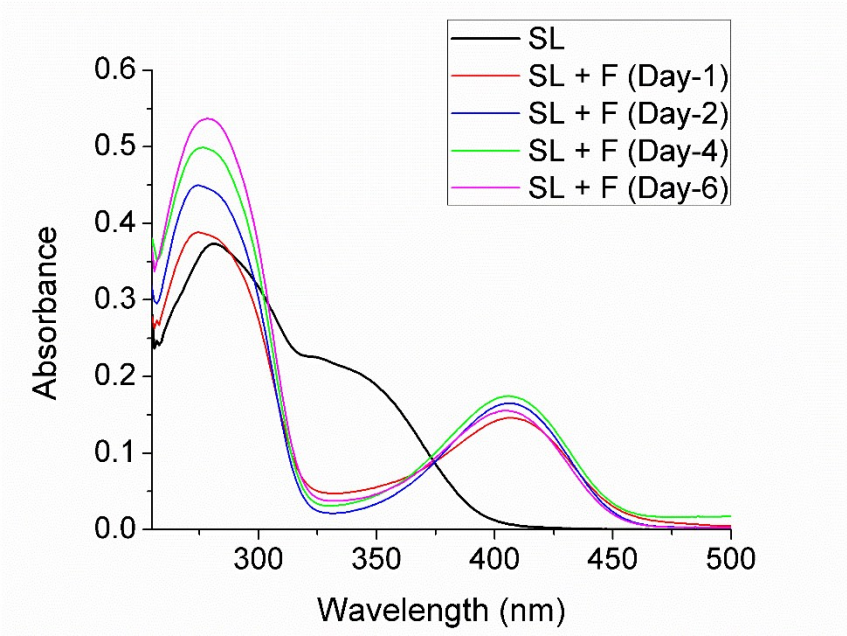


Fig. S37. UV-vis spectra of **SL** (1x10^{-5} mol/L) in the presence of TBAF (10 equiv.) recorded over a period of 6 days.

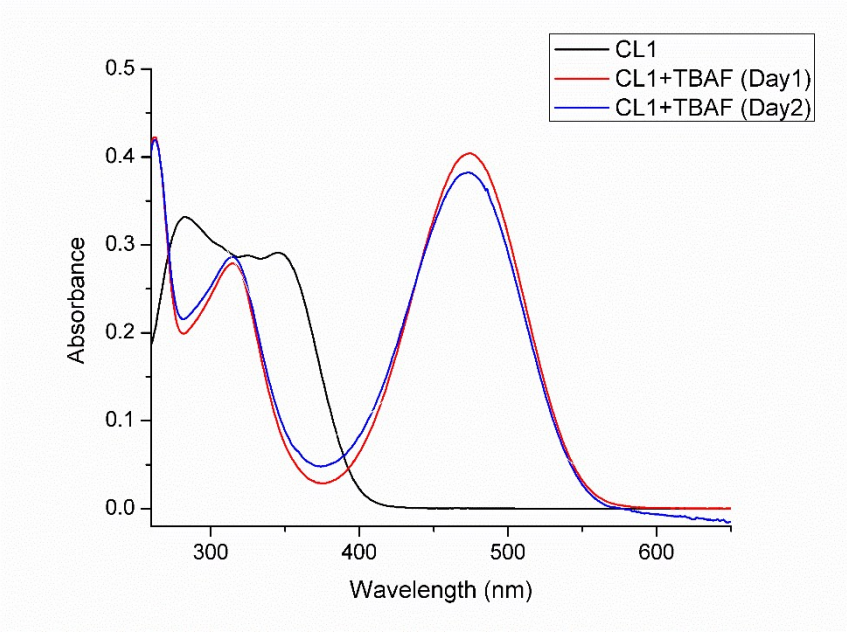


Fig. S38: UV-vis spectra of **CL1** ($1x10^{-5}$ mol/L) in the presence of TBAF (10 equiv.) recorded over a period of 2 days.

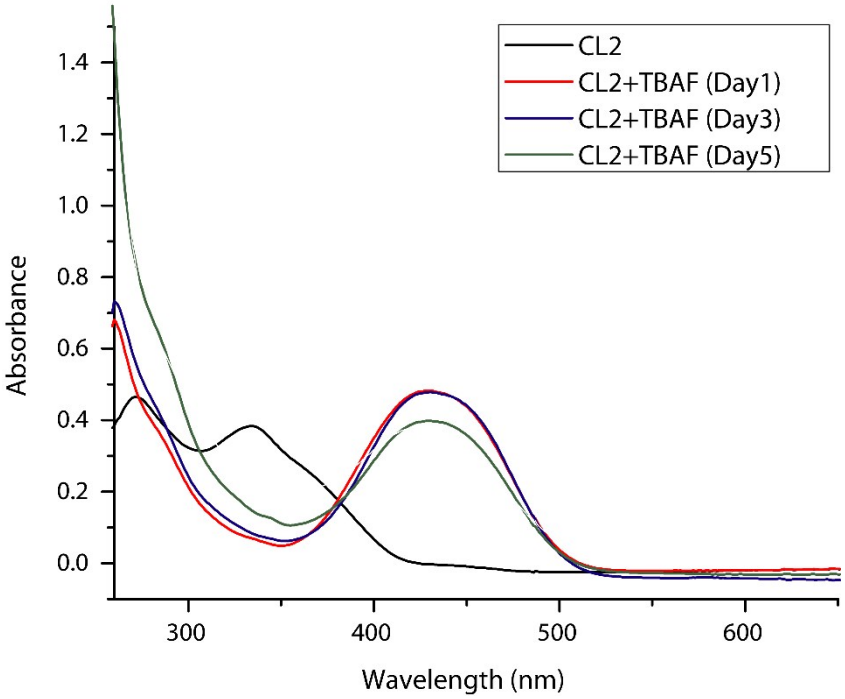


Fig. S39: UV-vis spectra of **CL2** ($1x10^{-5}$ mol/L) in the presence of TBAF (10 equiv.) recorded over a period of 5 days.

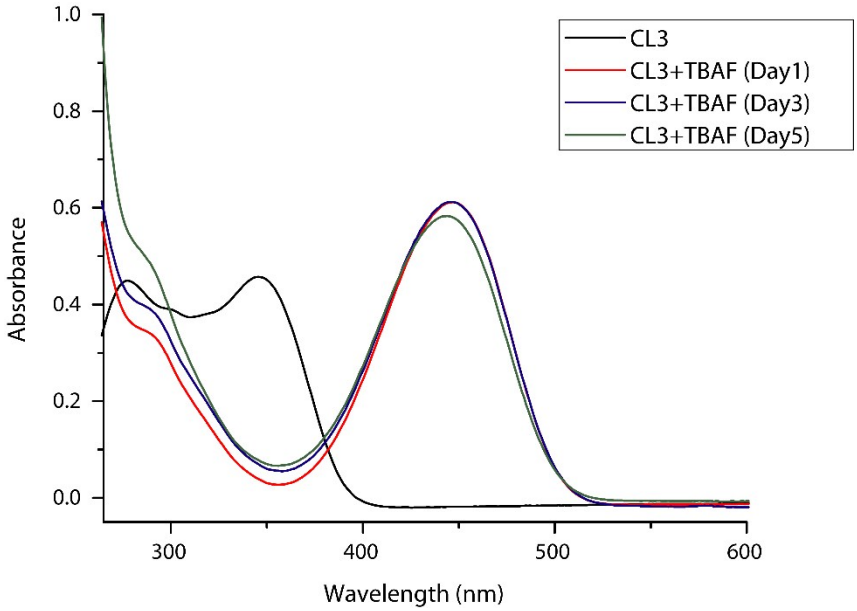


Fig. S40. UV-vis spectra of **CL3** ($1x10^{-5}$ mol/L) in the presence of TBAF (10 equiv.) recorded over a period of 5 days.

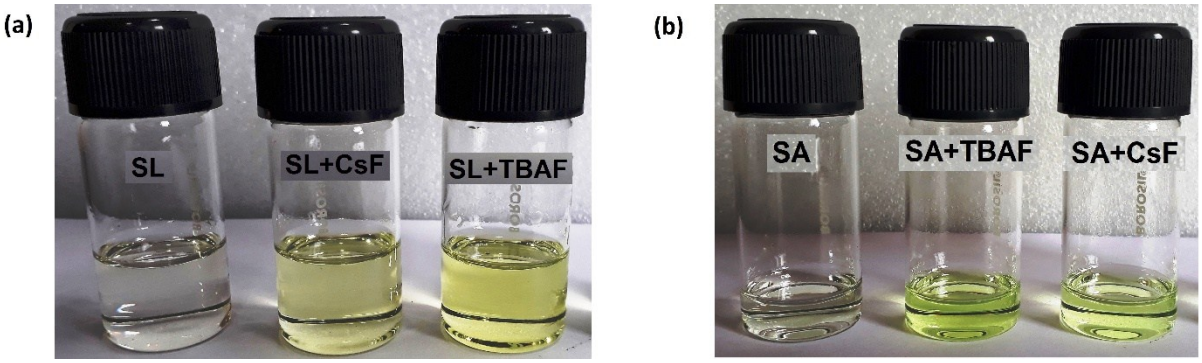


Fig. S41: (a) Colour changes of DMSO solutions ($1x10^{-4}$ M) of **SL** in the presence of 10 equivalents of CsF and TBAF, and (b) Colour changes of DMSO solutions ($1x10^{-4}$ M) of **Salicylaldehyde (SA)** in the presence of 10 equivalents of CsF and TBAF.