

Preparation and Characterization of a Novel Silica-KF Composite and Facile Fluorination of Aromatic Substrates

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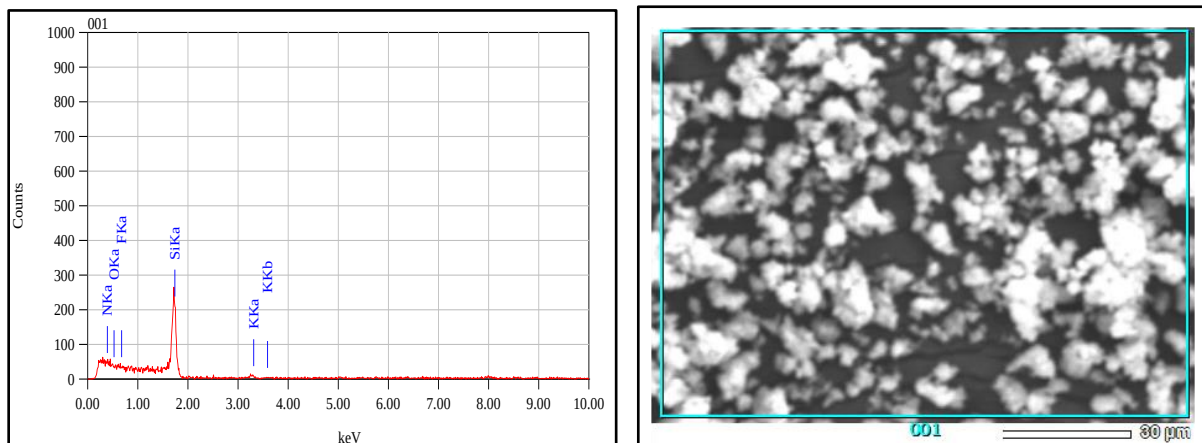
1. Moisture content

Time (Hrs)	Change in weight KF	% Change in weight KF	Change in weight Silica-KF	% Change in weight Silica-KF
0	0.000	0.000	0.000	0.000
1	0.108	5.118	0.069	4.542
2	0.185	8.459	0.087	5.660
3	0.271	11.923	0.110	7.051
4	0.364	15.385	0.118	7.526
5	0.447	18.252	0.119	7.584
6	0.530	20.932	0.126	7.995
7	0.573	22.252	0.131	8.286
24	1.410	41.325	0.184	11.261
48	1.708	46.038	0.189	11.531

S 1: Moisture Uptake Capacity of KF and silica-KF Raw data

2. SEM-EDX analysis

EDX results for silica-KF reagent showing excess of fluoride anion mol% on the surface.



ZAF Method Standardless Quantitative Analysis(Oxide)

Fitting Coefficient: 0.7473

Total Oxide: 24.0

Element	(keV)	mass%	Error%	Mol%	Compound	mass%	Cation	K
H K								
N K	0.392	0.38	2.87	1.57	N	0.38	0.00	0.9133
O		51.01						
F K	0.677	2.30	2.03	6.90	F	2.30	0.00	0.9016
Si K	1.739	44.43	1.28	90.17	SiO ₂	95.06	11.91	93.9602
K K	3.312	1.88	1.05	1.37	K ₂ O	2.26	0.36	4.2249
Total		100.00		100.00		100.00	12.27	

S 2: SEM-EDX data for surface analysis of silica-KF

3. X- ray diffraction

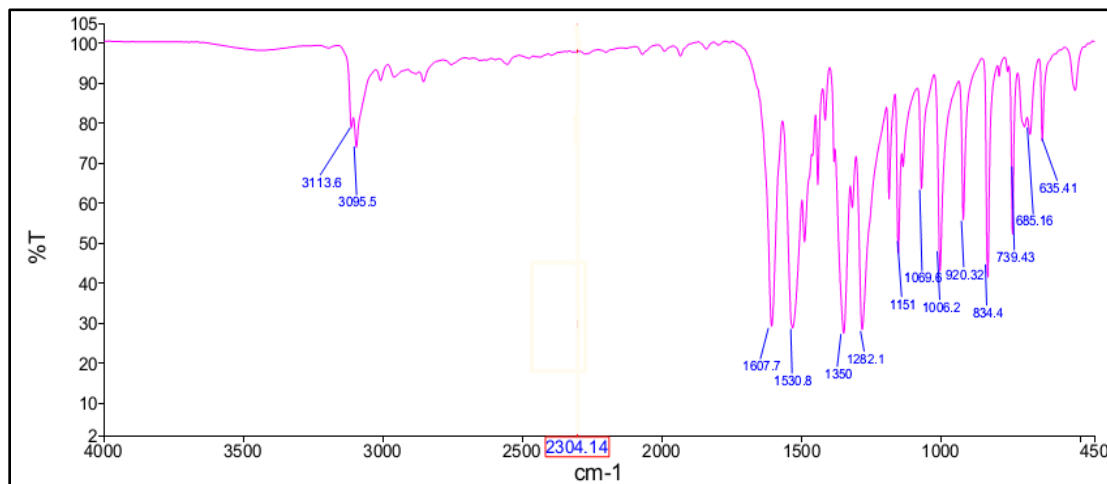
d values important peaks in the diffractogram of silica-KF and silica-KF washed with water is shown below.

SSPSKF			SSPSKFW		
2 Theta	d value	% intensity	2 Theta	d value	% intensity
18.086	4.90089	16	18.0406	4.91311	41.4
18.8824	4.69595	100	18.8547	4.70277	100
19.2632	4.60395	23.4	20.3114	4.30575	75.7
20.5216	4.3244	40.2	31.0541	2.87755	56.2
26.5463	3.35506	59.5	36.6208	2.4519	13
31.0742	2.87573	64.2	38.2935	2.34856	43.8
31.6143	2.82782	25.4	39.0498	2.30479	10.9
34.4531	2.60104	12.3	41.7359	2.16245	14
36.6255	2.45159	10.2	44.5118	2.03383	31.5
38.2934	2.34856	55.2			
39.0891	2.30257	12.2			
41.7333	2.16259	31.4			
44.5053	2.03411	40.6			
47.4211	1.91561	12.6			

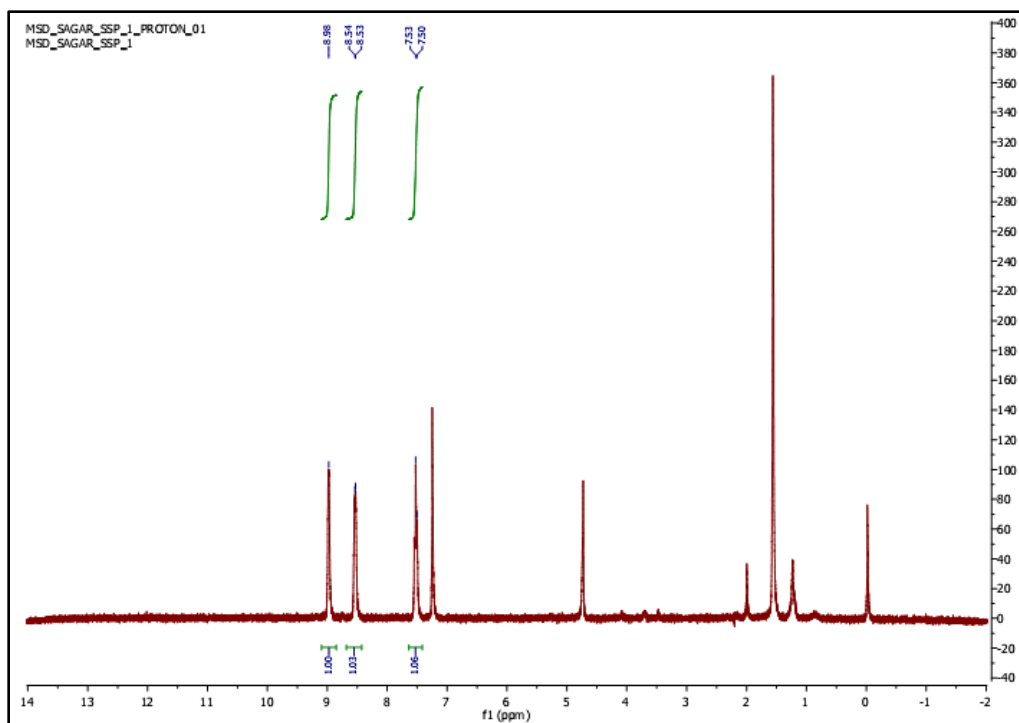
S 3: d values for silica-KF(SSPKF), silica-KF(SSPKFW) washed with water

4. Characterization of compounds

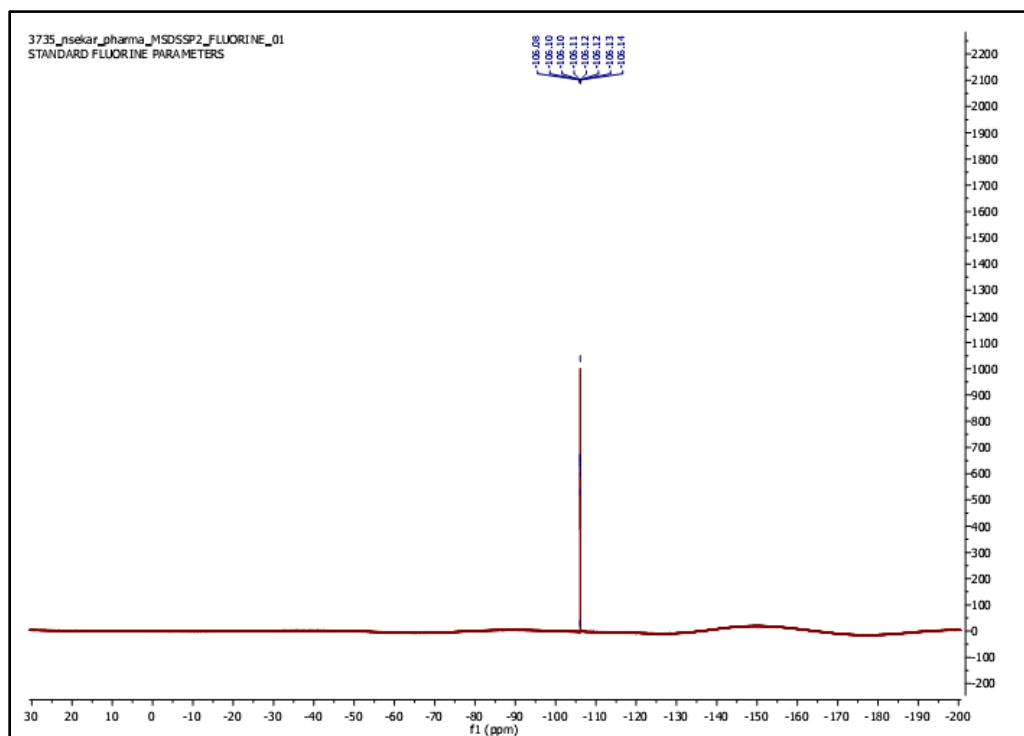
2,4 DNFB



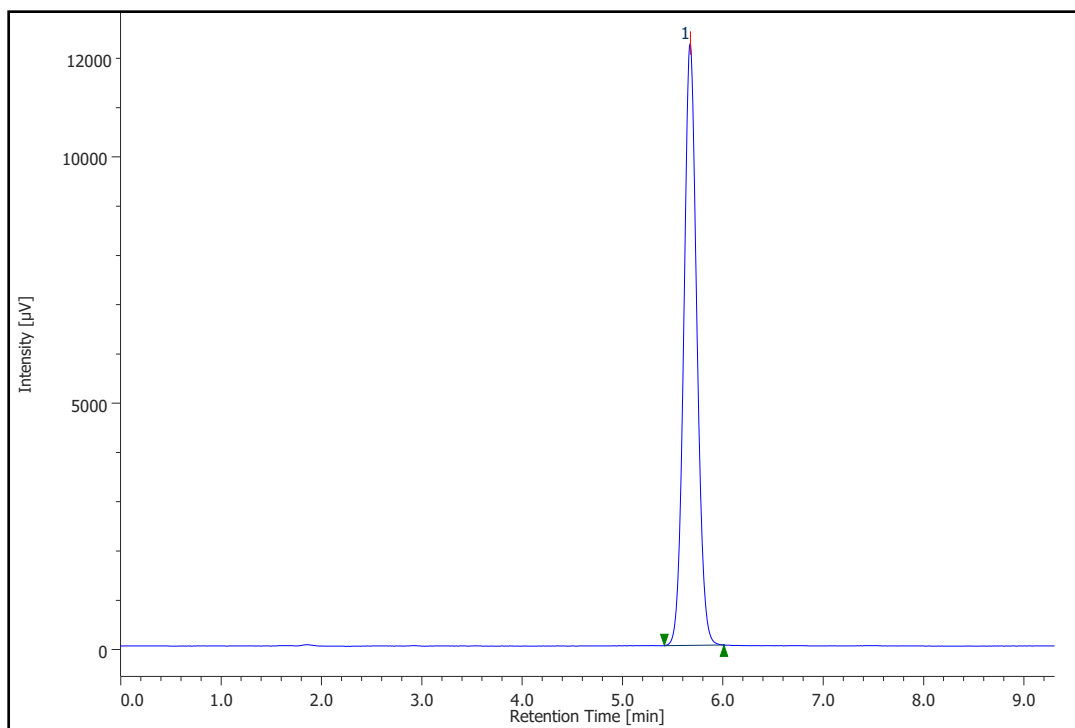
S 4: IR spectra of DNFB



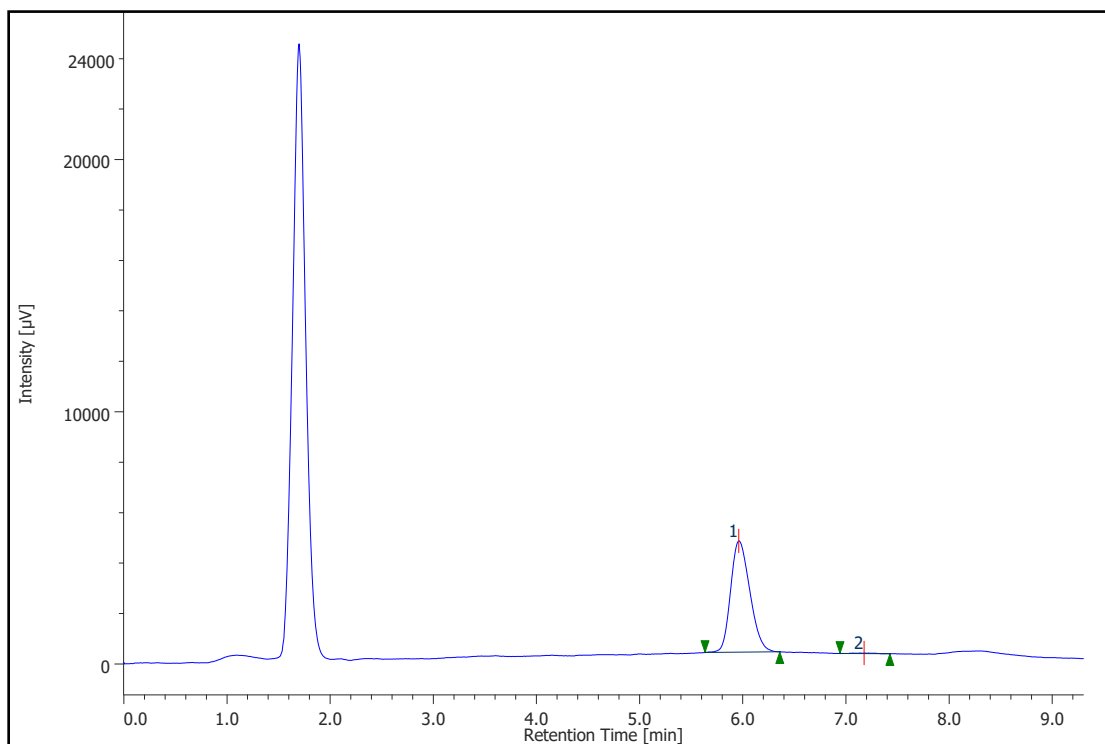
S 5: ^1H NMR spectra of DNFB



S 6: ^{19}F NMR spectra of 2,4 DNFB

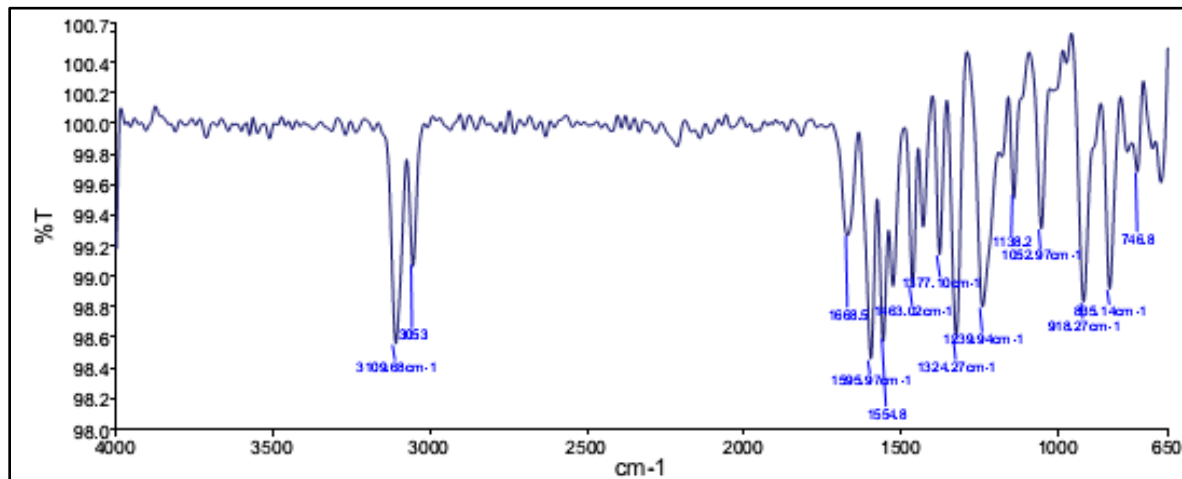


S 7: HPLC chromatogram of 2,4 DNFB

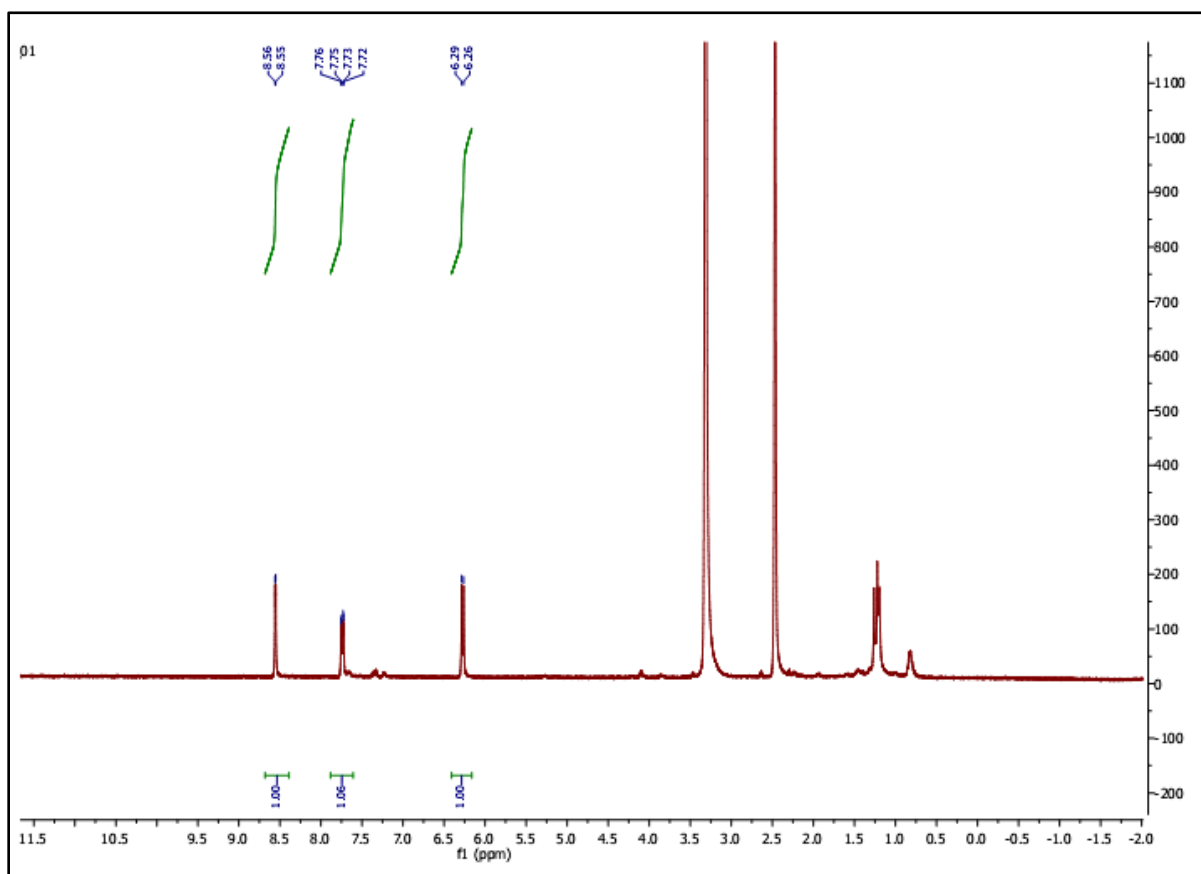


S 8: Reaction mass of 2,4 DNFB with silica-KF

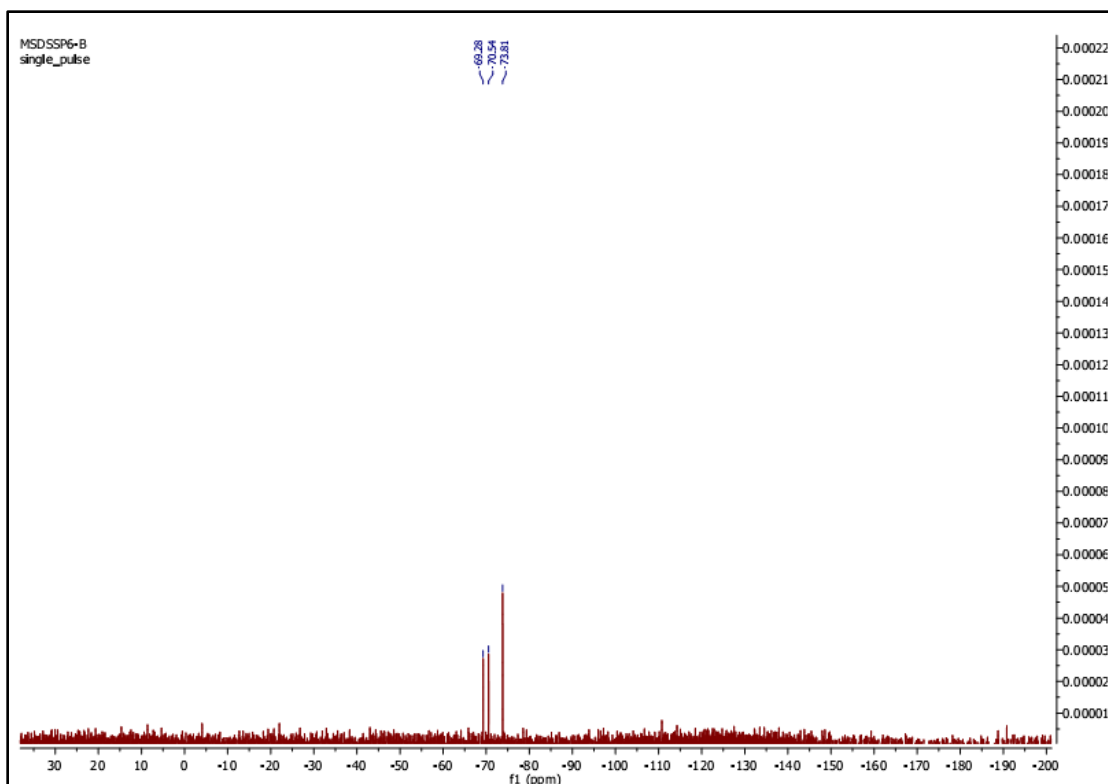
Meisenheimer complex of 2,4 DNCB with fluoride ion



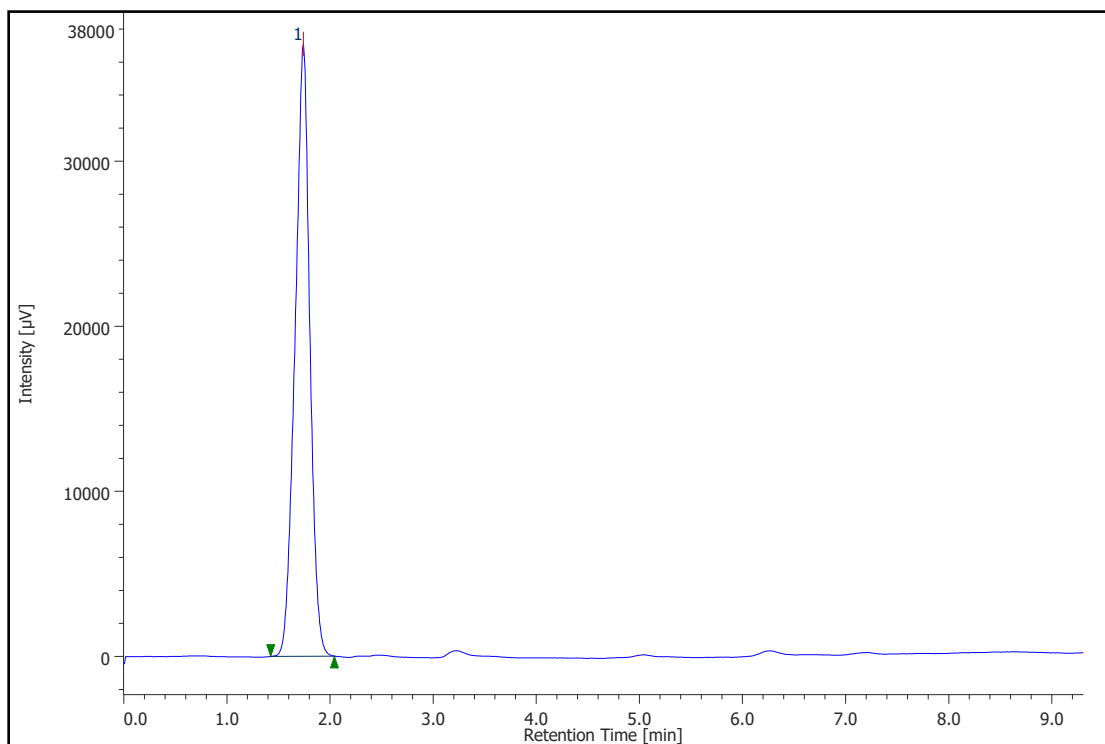
S 9: IR spectra of Meisenheimer complex of 2,4 DNCB



S 10: ^1H NMR spectra of Meisenheimer complex of 2,4 DNCB

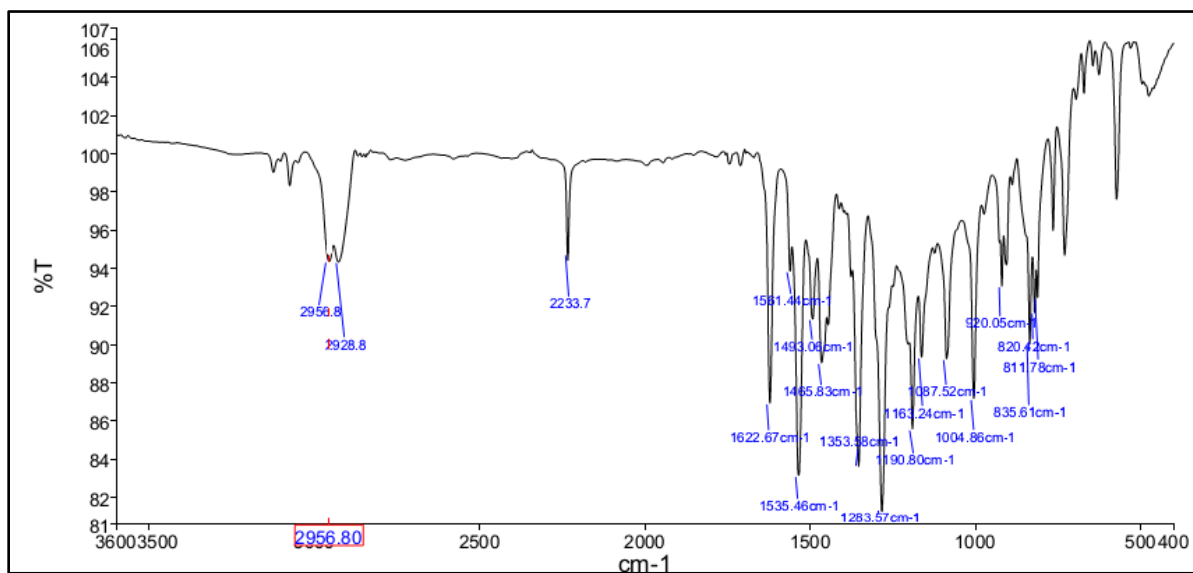


S 11: ^{19}F NMR of Meisenheimer complex of 2,4 DNCB

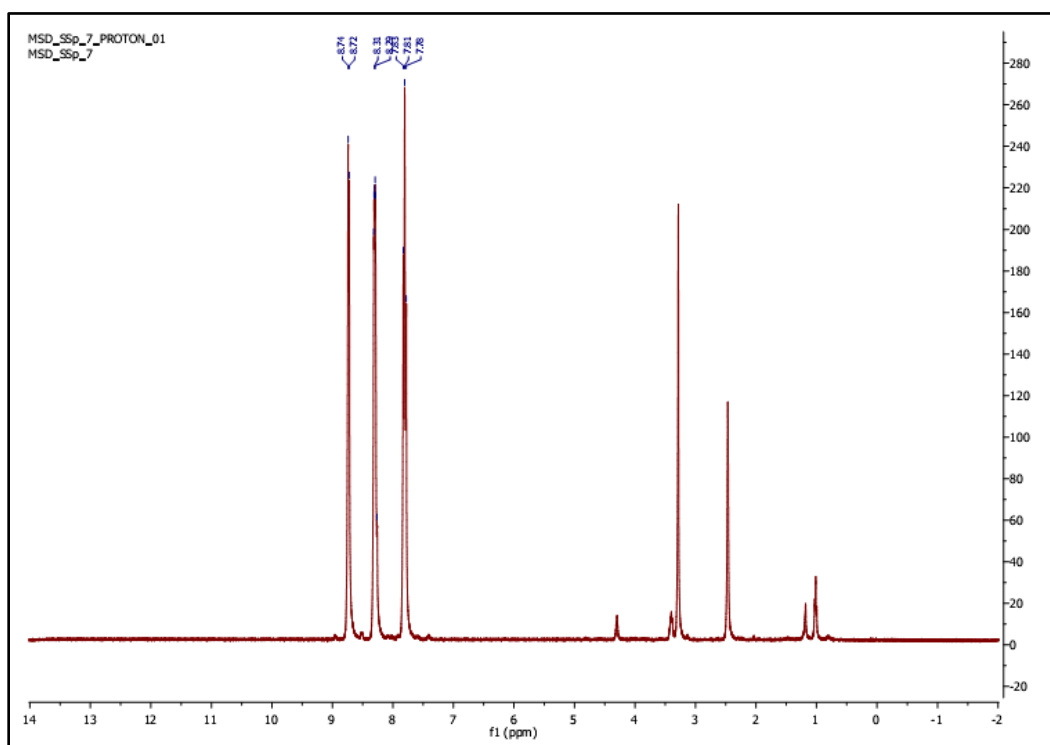


S 12: HPLC chromatogram of Meisenheimer complex of 2,4 DNCB

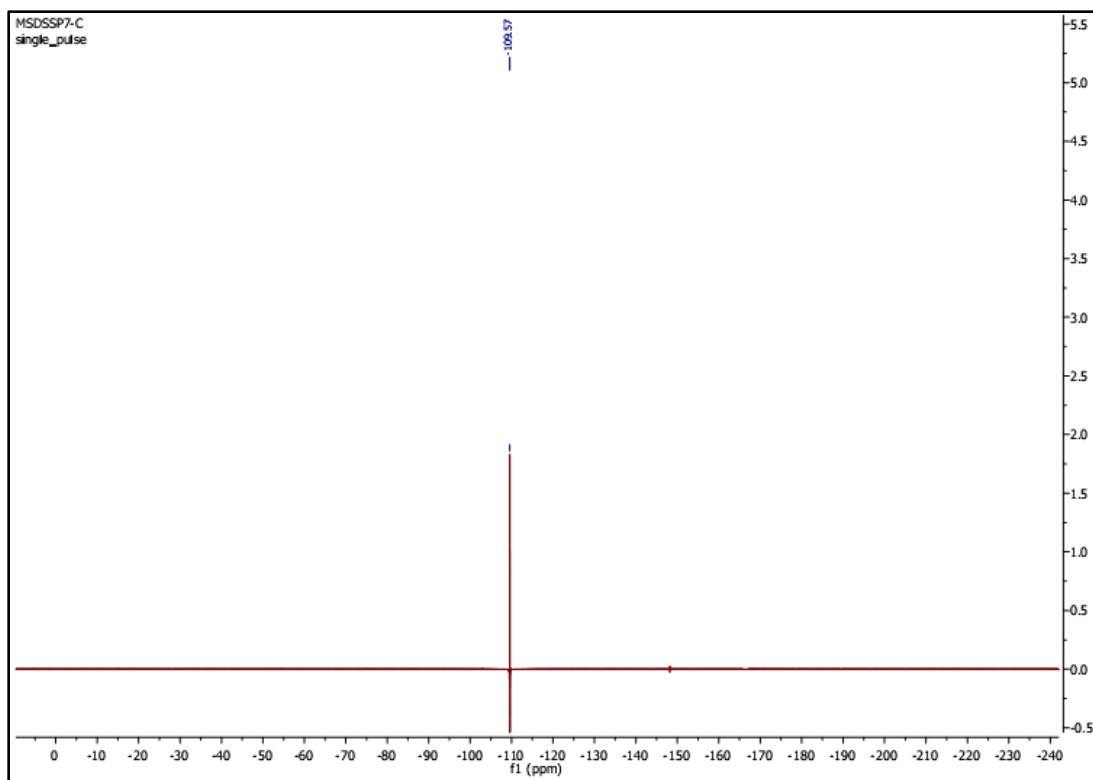
4-fluoro 3-nitrobenzonitrile(4F3NBn)



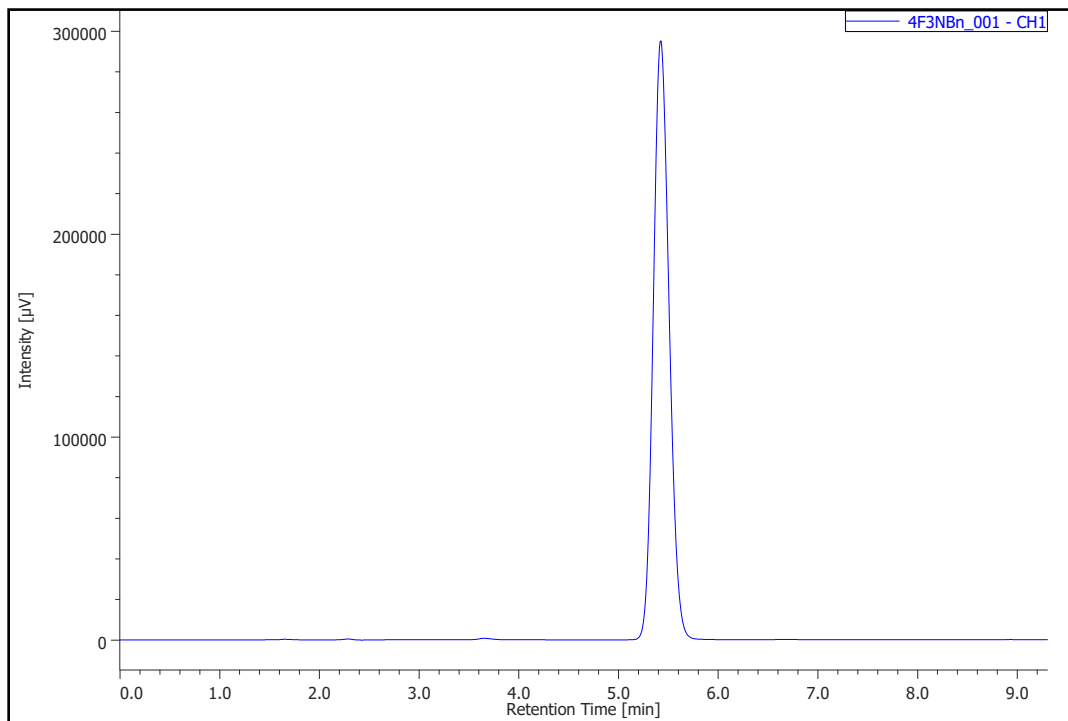
S 13: IR spectra of 4F3NBn



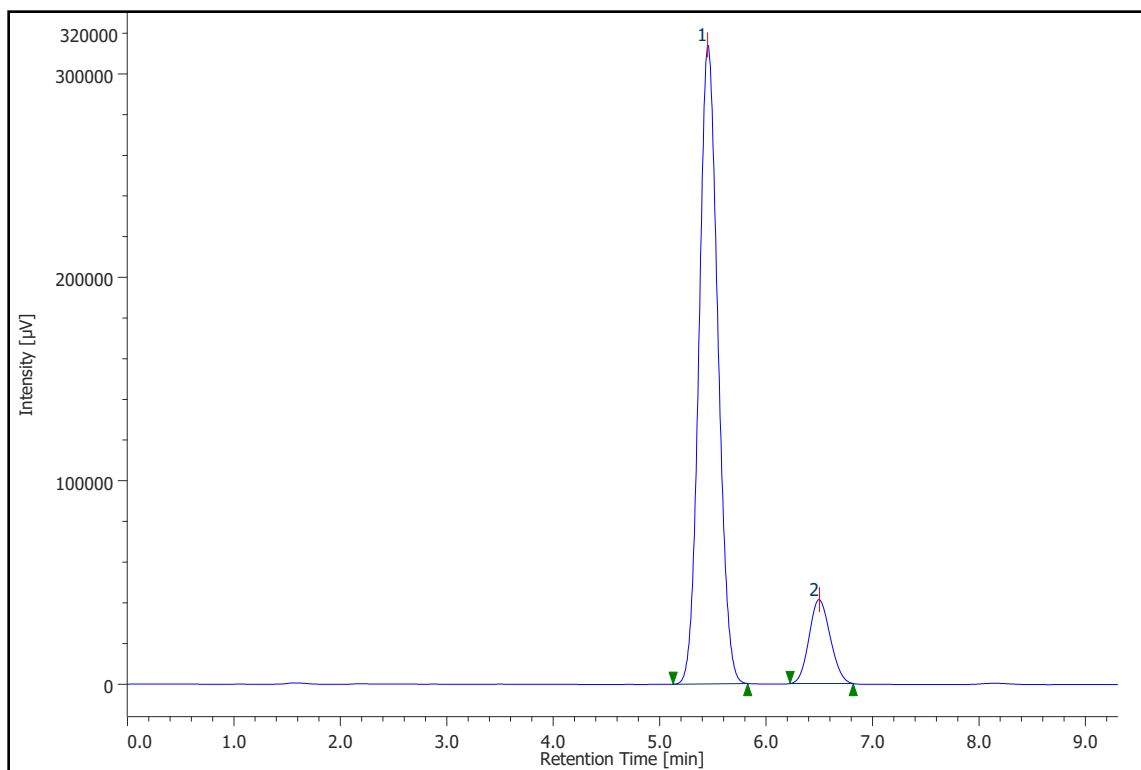
S 14: ^1H NMR of 4F3NBn



S 15: ^{19}F NMR of 4F3NBn

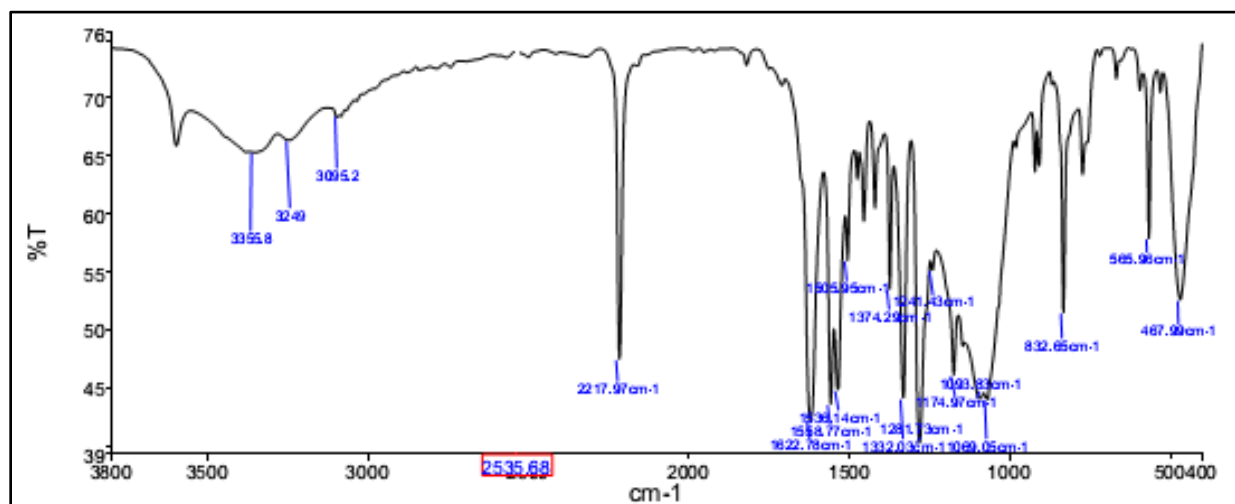


S 16: HPLC chromatogram of 4F3NBn

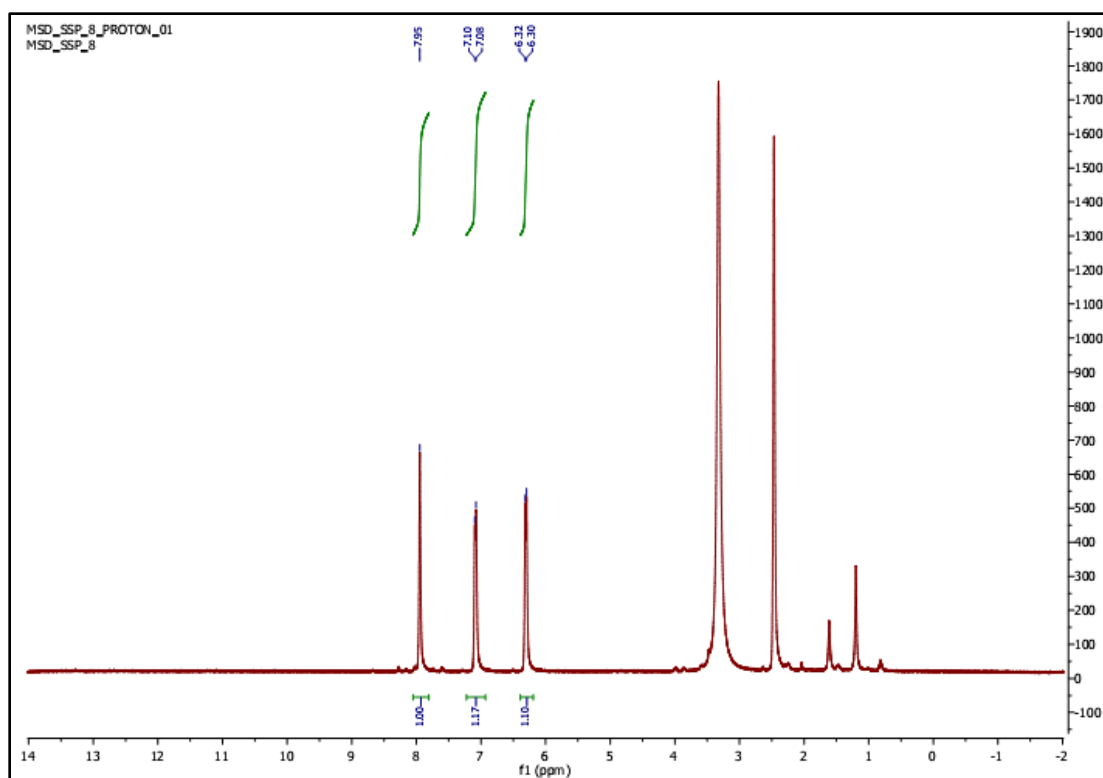


S 17: Reaction of 4C3NBn with silica-KF

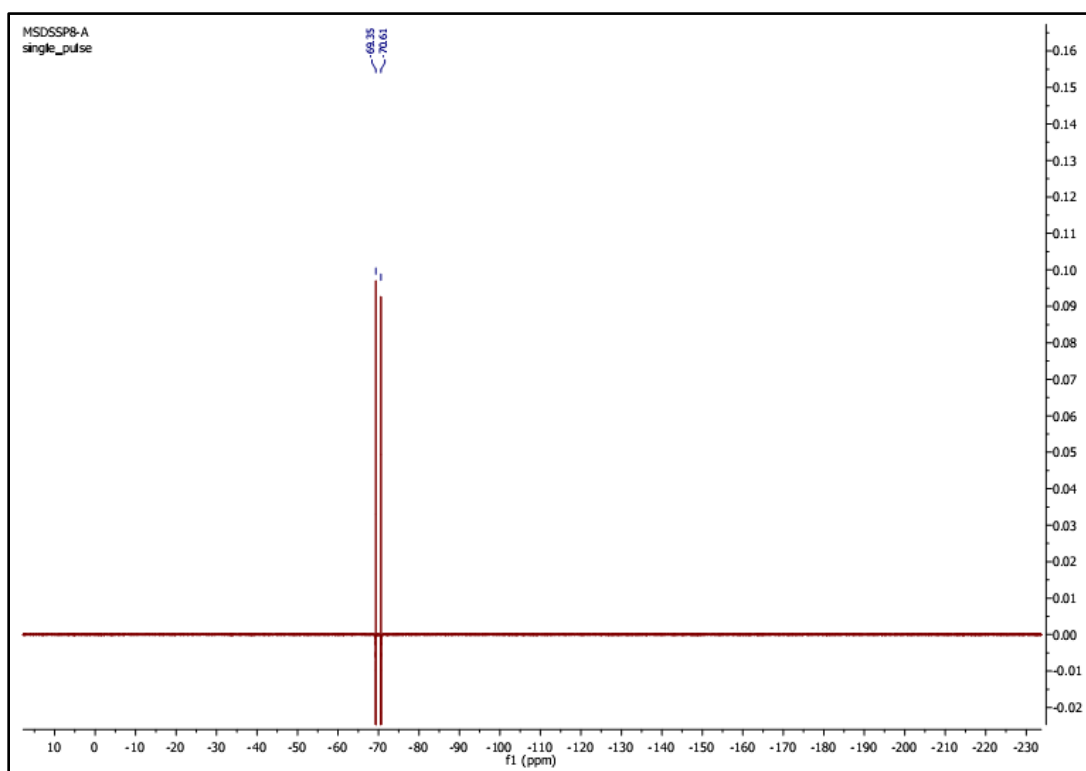
Meisenheimer complex of 4F3NBn with chloride ion



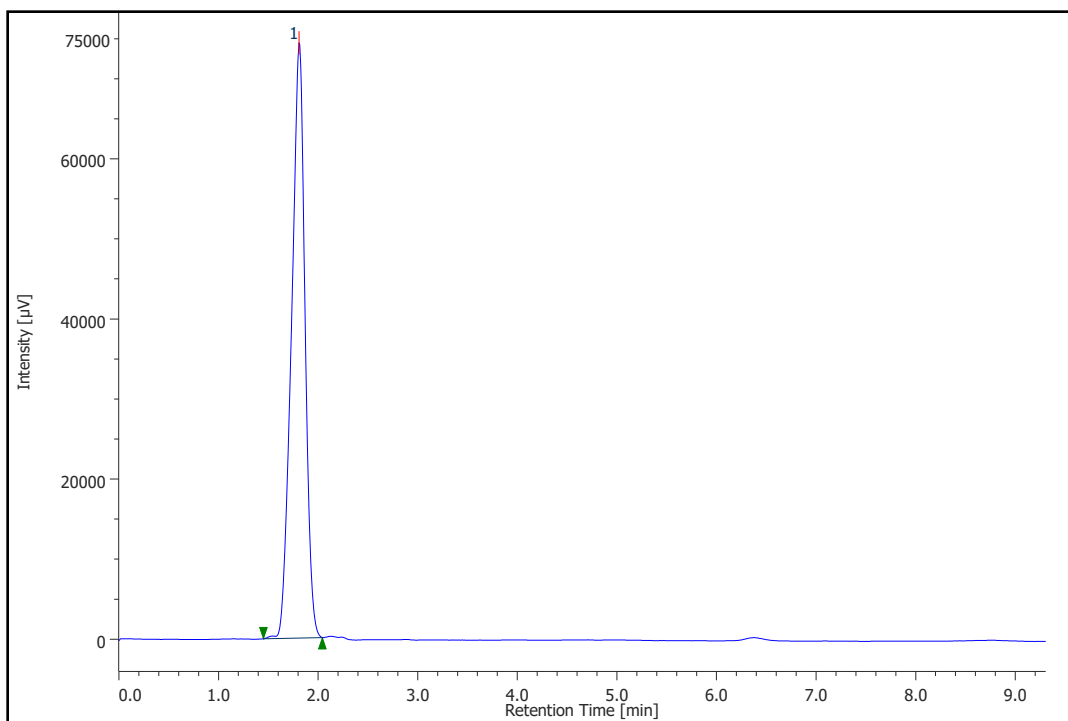
S 18: IR spectra of Meisenheimer complex of 4Cl3NBn



S 19: ^1H NMR spectra of Meisenheimer complex of 4Cl3NBn

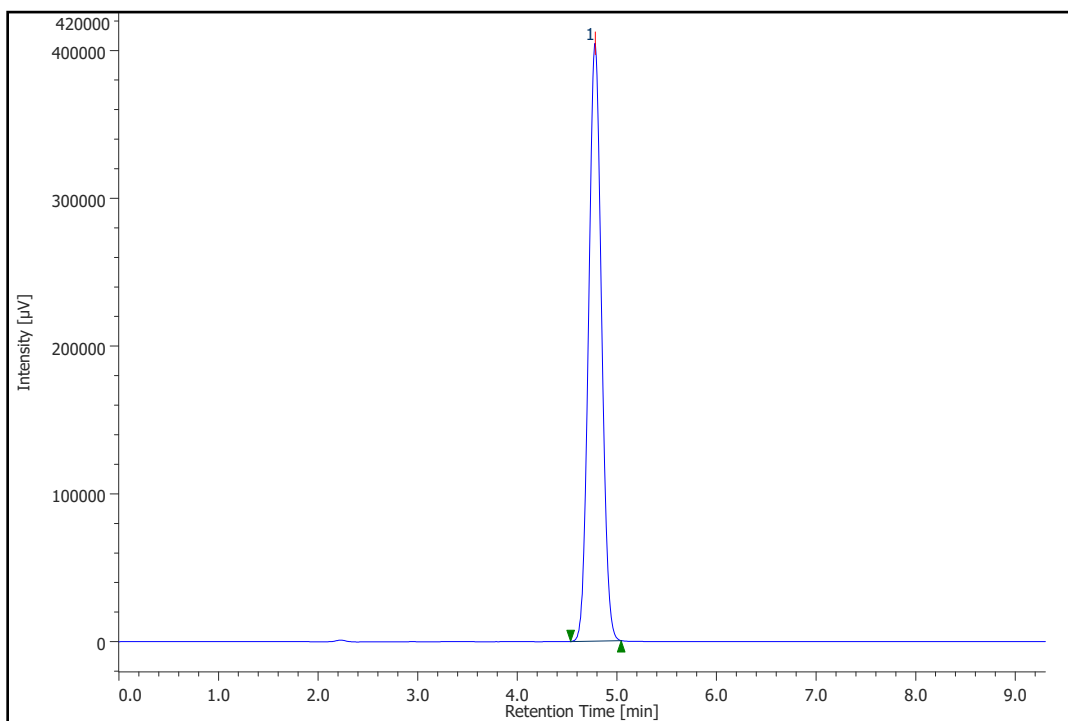


S 20: ^{19}F NMR spectra of Meisenheimer complex of 4Cl3NBn

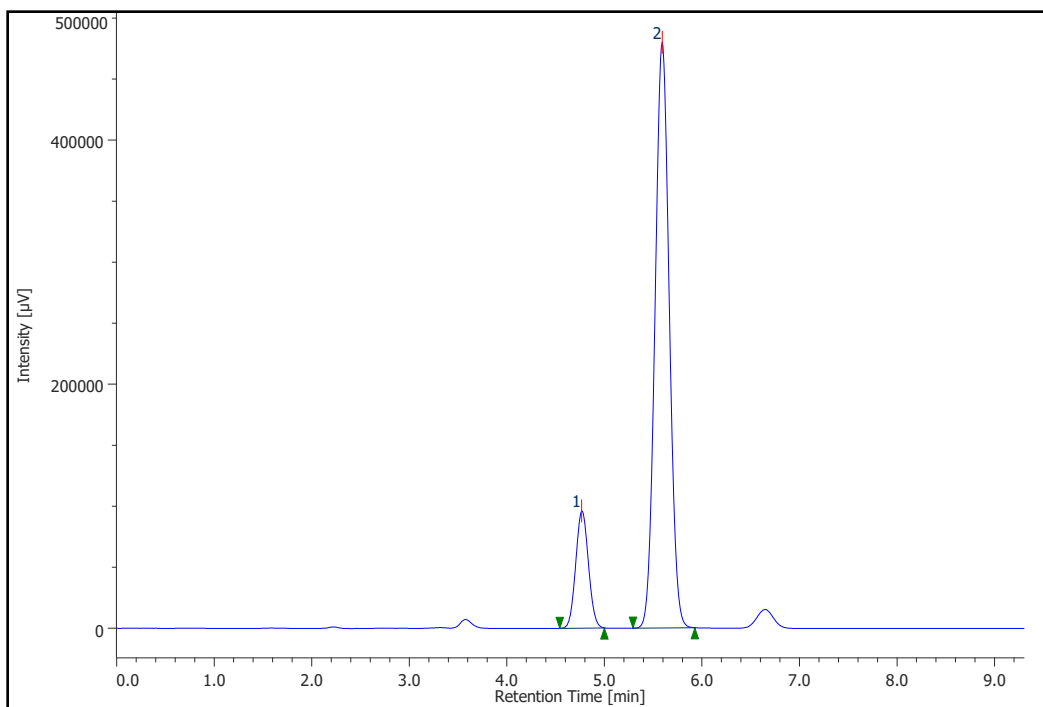


S 21: HPLC chromatogram of Meisenheimer complex of 4Cl₃NBn

4-fluoro nitrobenzene (4-FNB)

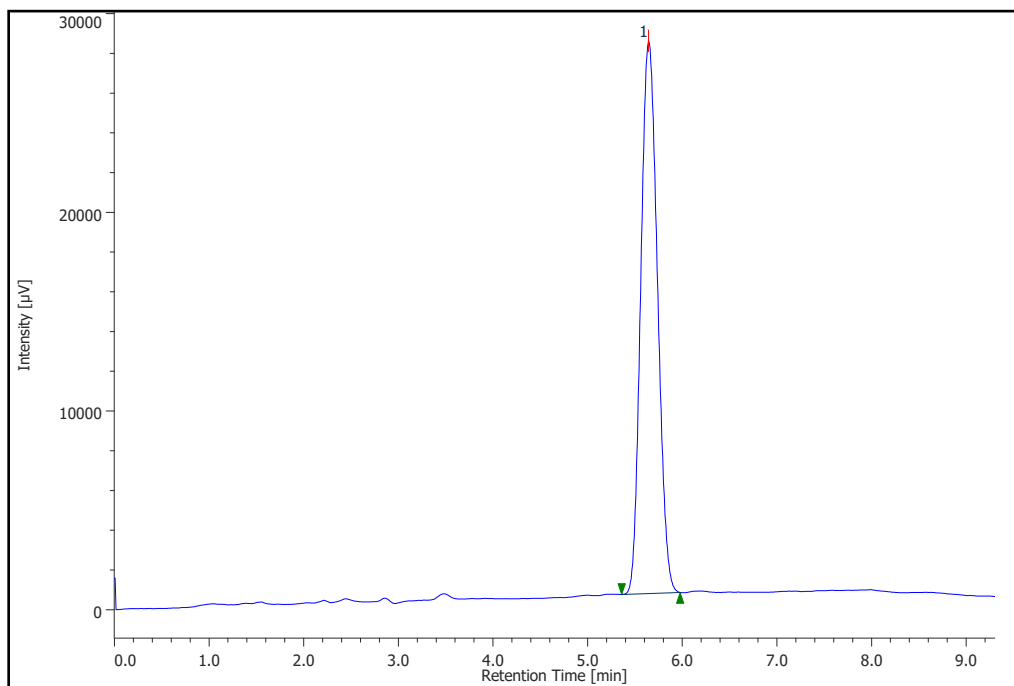


S 22: HPLC chromatogram of standard 4-fluoronitrobenzene

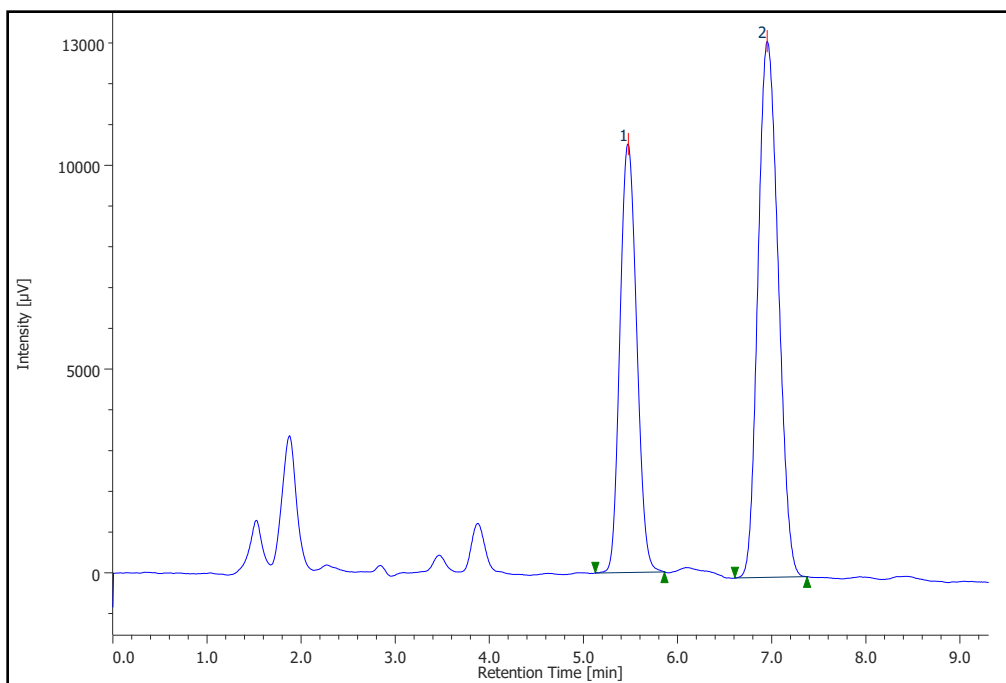


S 23: Reaction of 4-chloro nitrobenzene with silica-KF

4-fluoro benzonitrile (4-FBn)



S 24: HPLC chromatogram of standard 4-FBn



S 25: Reaction of 4-chloro nitrobenzene with silica-KF