

Supporting Information

Triphenylphosphine oxide removal from reaction: Role of solvent and temperature

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Figure S1: Compound-3: ^1H NMR, DMSO-d6, 400 MHz

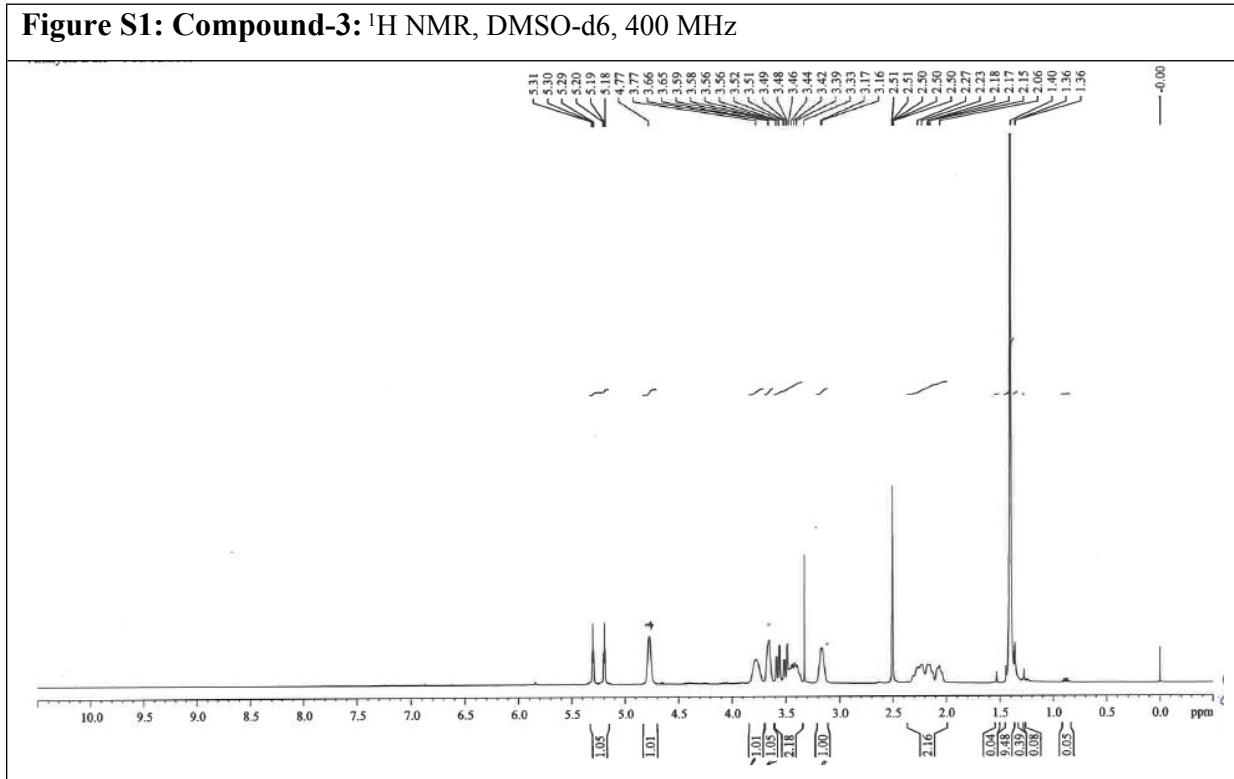
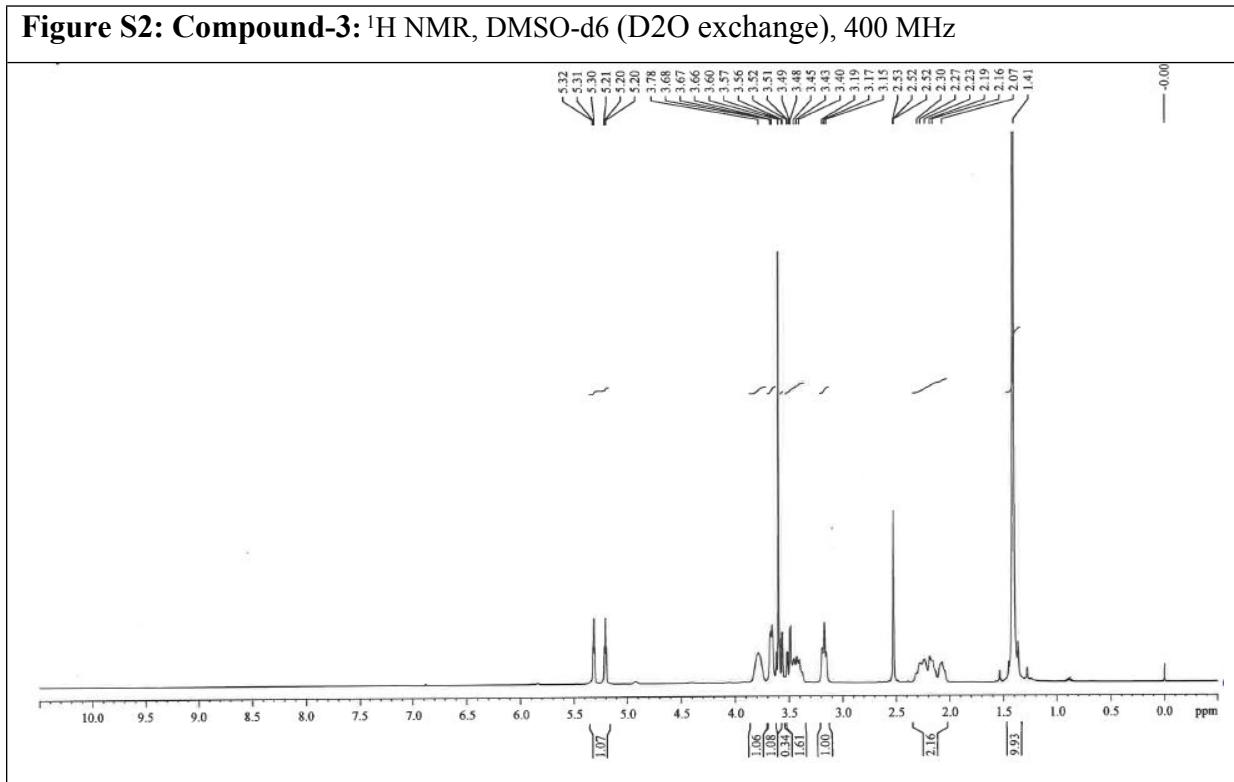
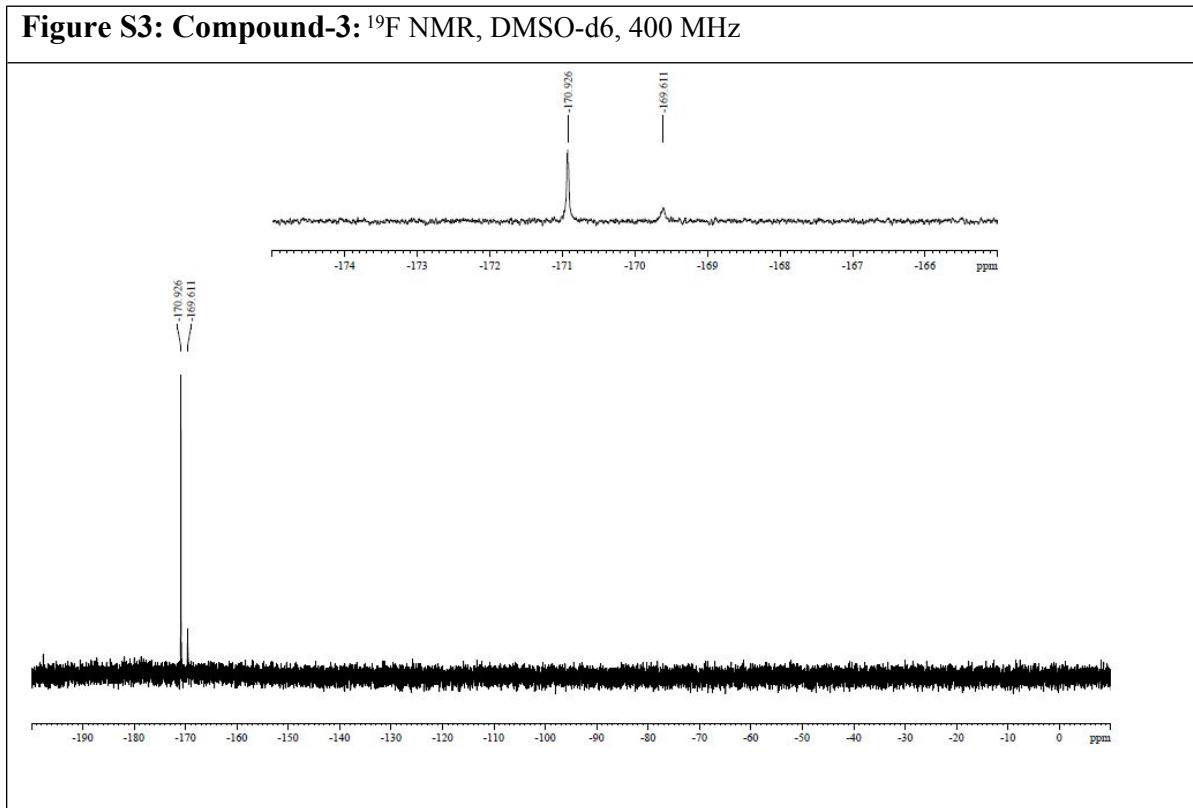


Figure S2: Compound-3: ^1H NMR, DMSO-d6 (D₂O exchange), 400 MHz



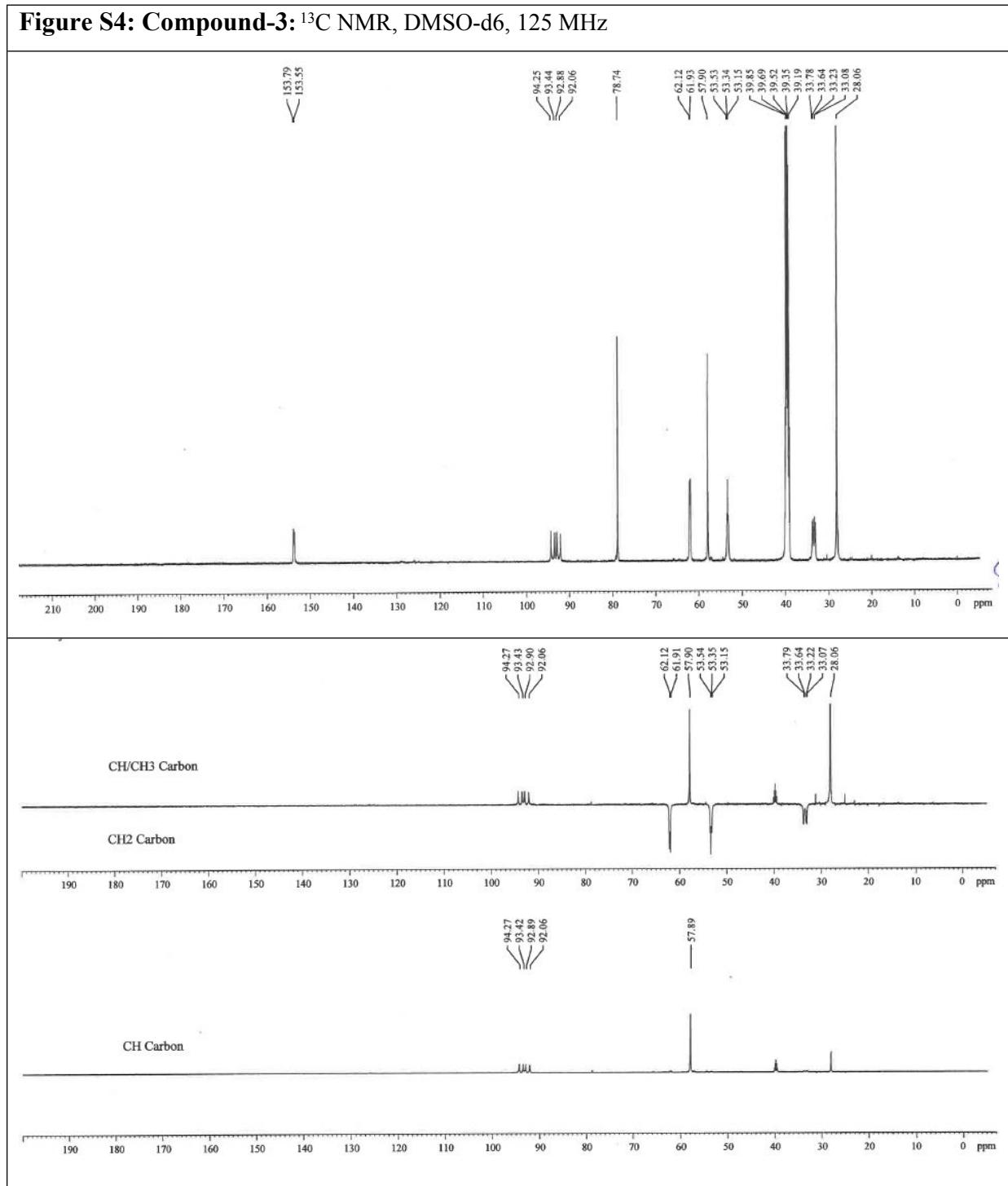
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Figure S3: Compound-3: ^{19}F NMR, DMSO-d₆, 400 MHz



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Figure S4: Compound-3: ^{13}C NMR, DMSO-d₆, 125 MHz



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Figure S5: Compound-3: Mass

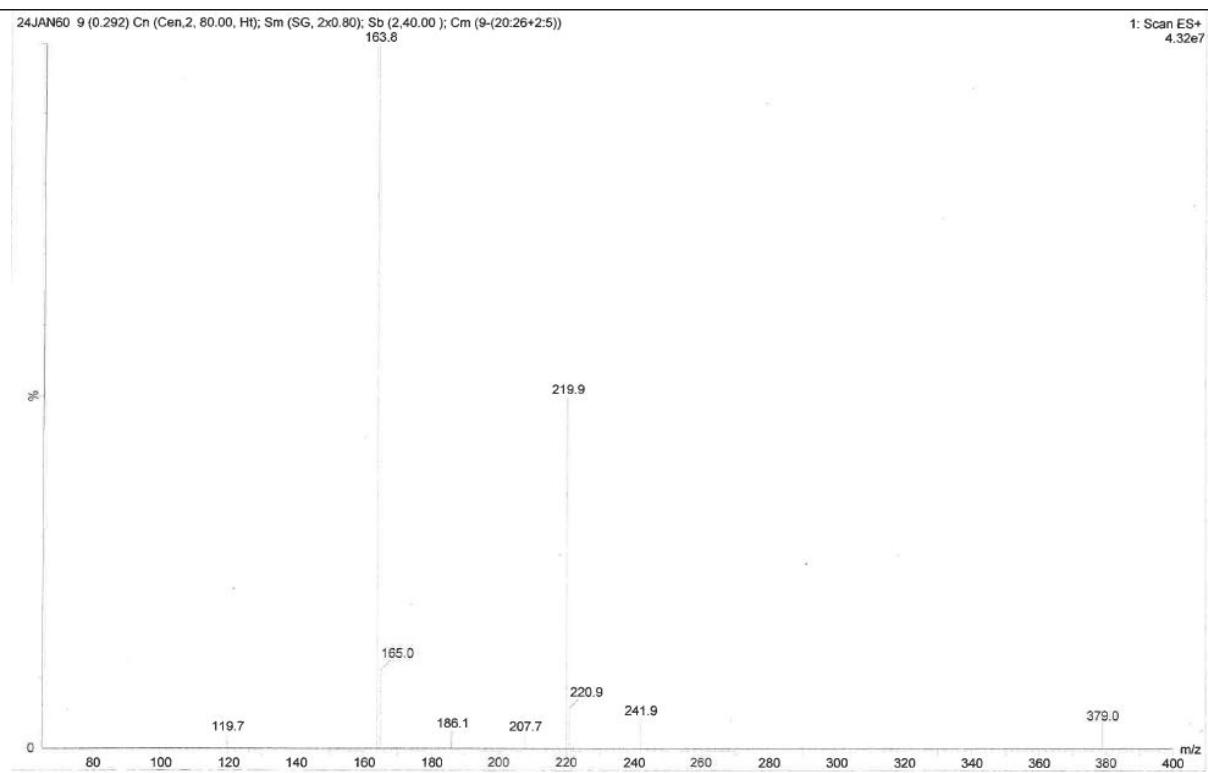
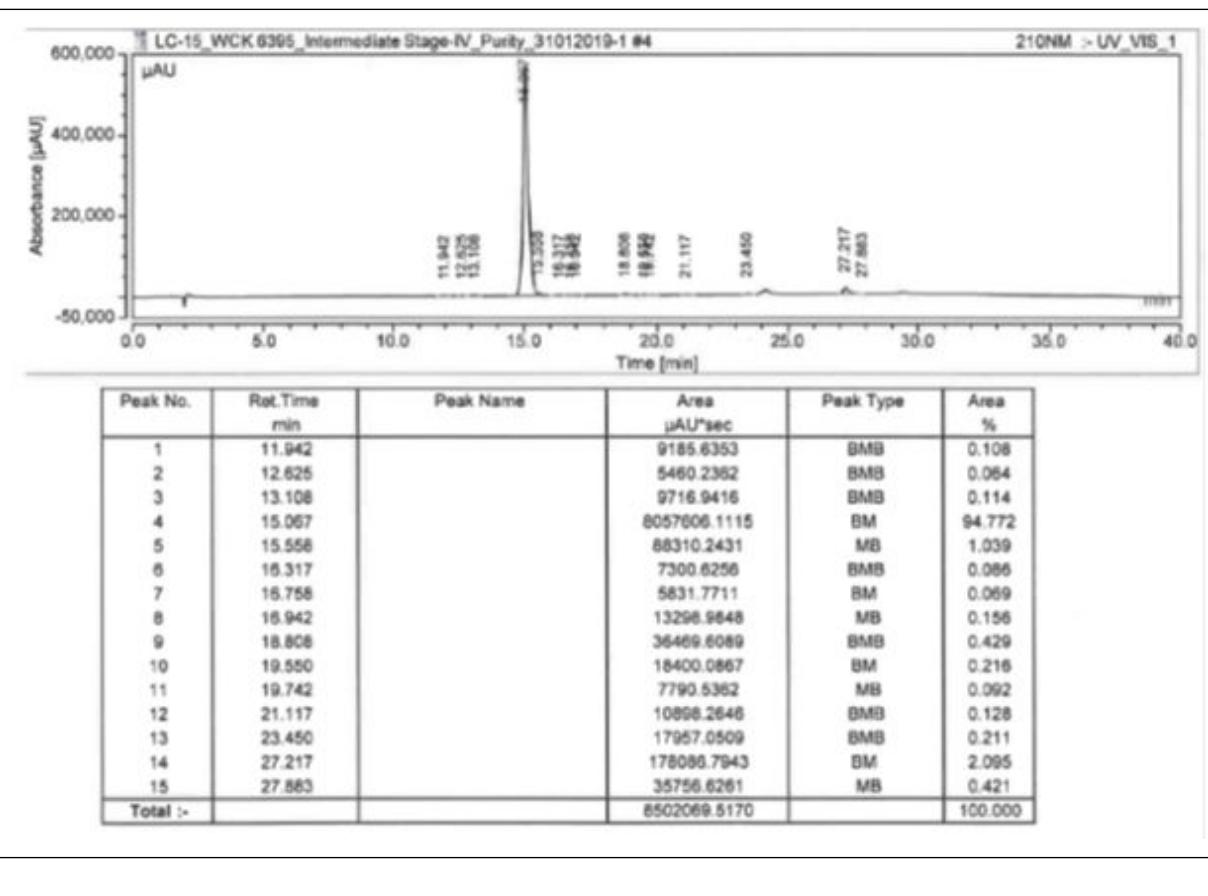


Figure S6: Compound-3: HPLC



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Figure S7: Compound-6: ^1H NMR, CDCl_3 , 400 MHz

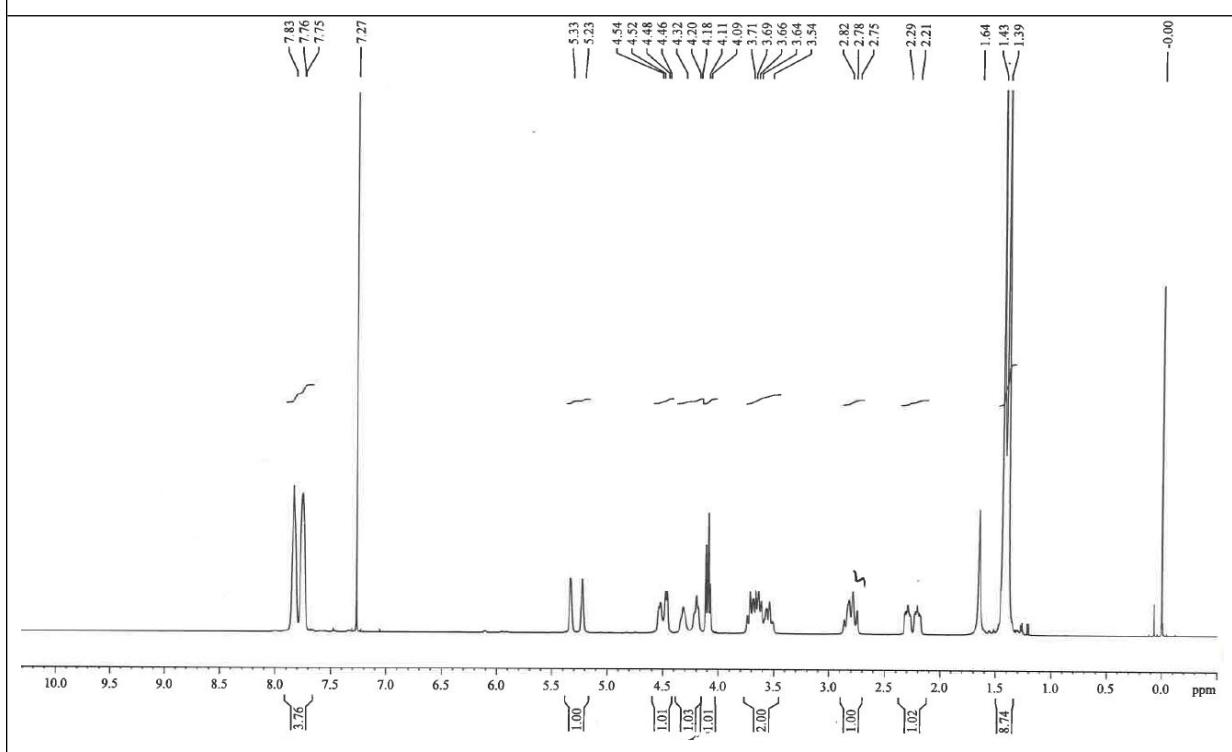
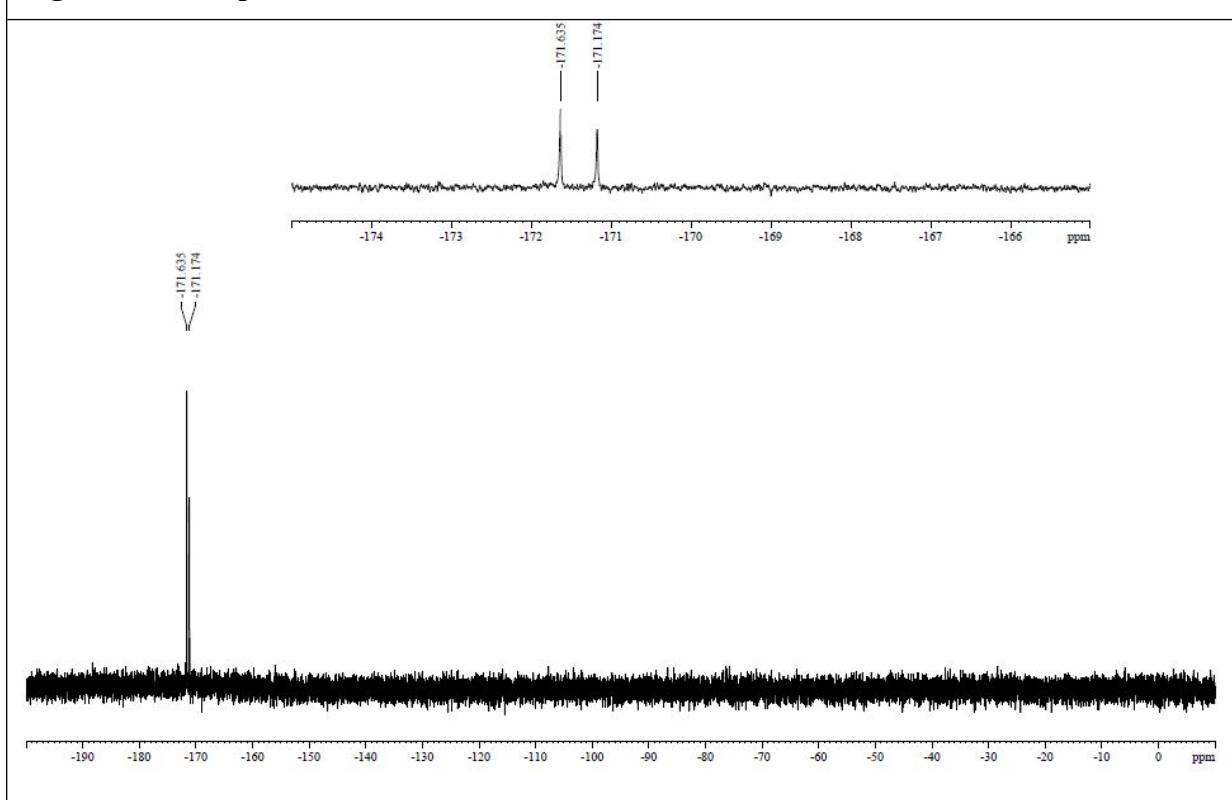
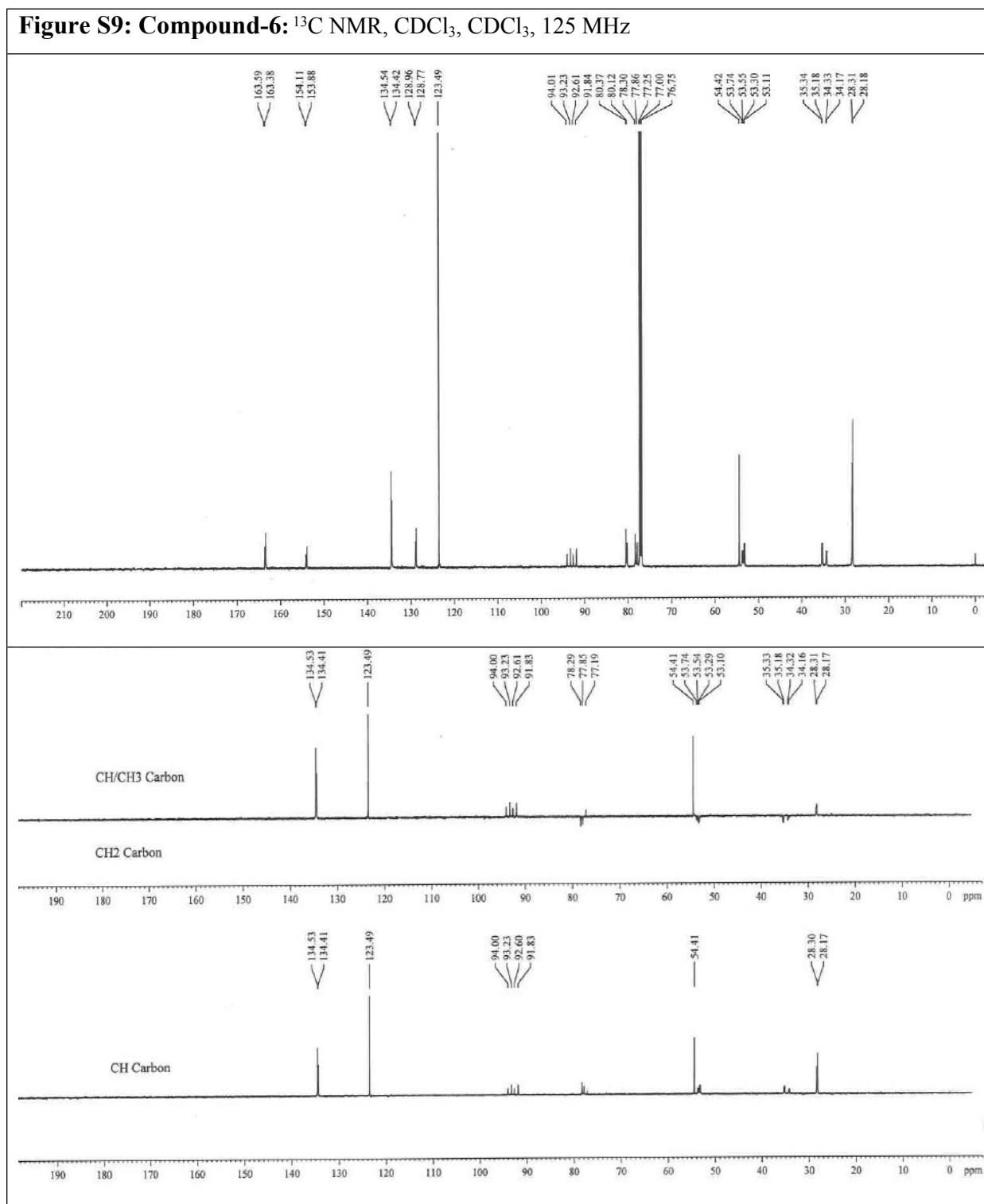


Figure S8: Compound-6: ^{19}F NMR, CDCl_3 , 400 MHz



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Figure S9: Compound-6: ^{13}C NMR, CDCl_3 , CDCl_3 , 125 MHz



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Figure S10: Compound-6: Mass

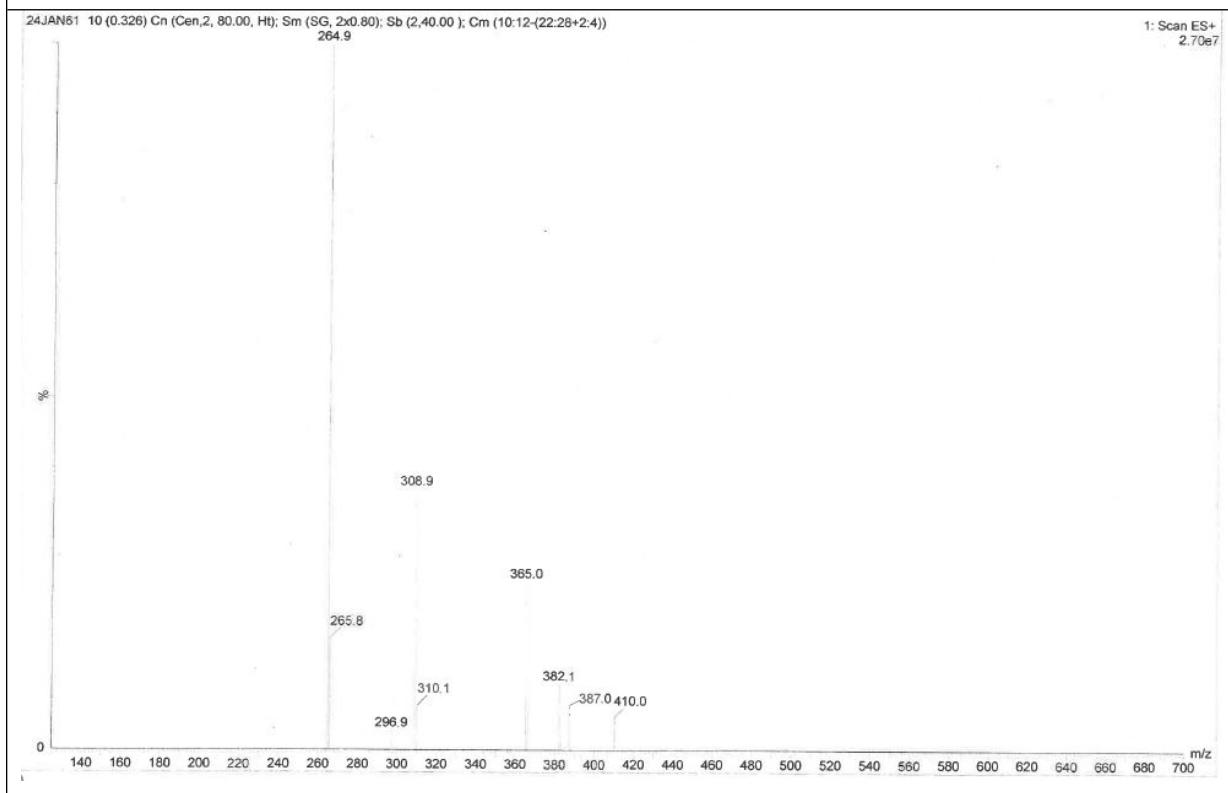
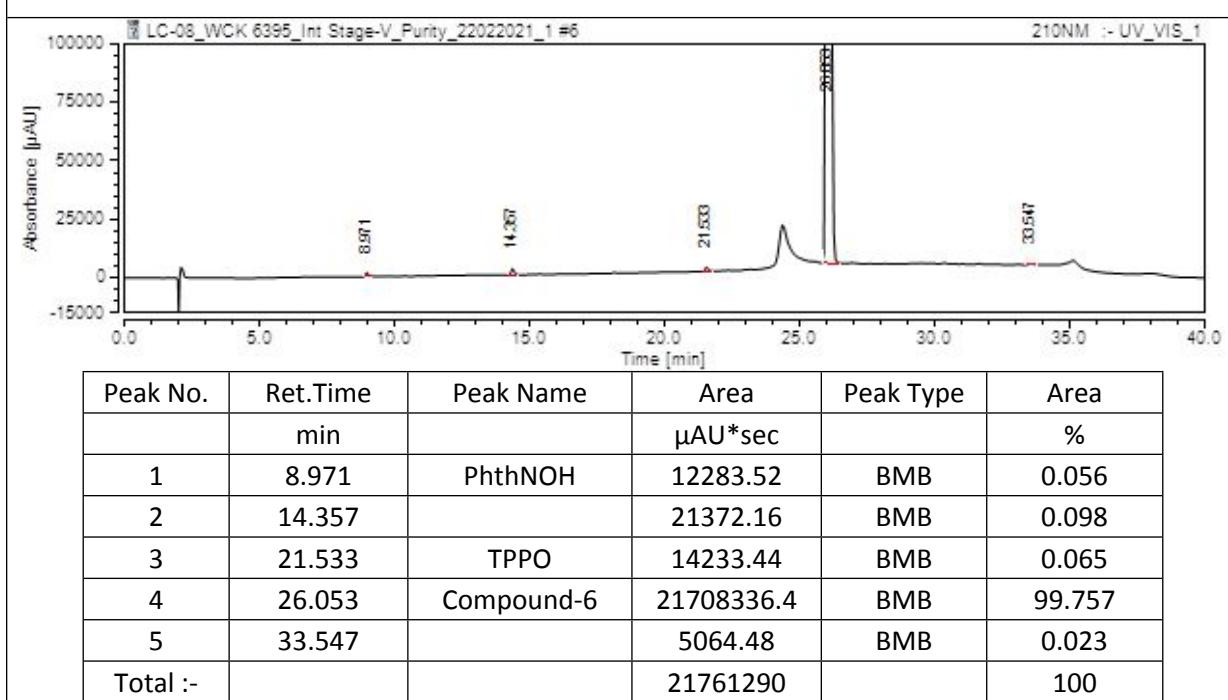
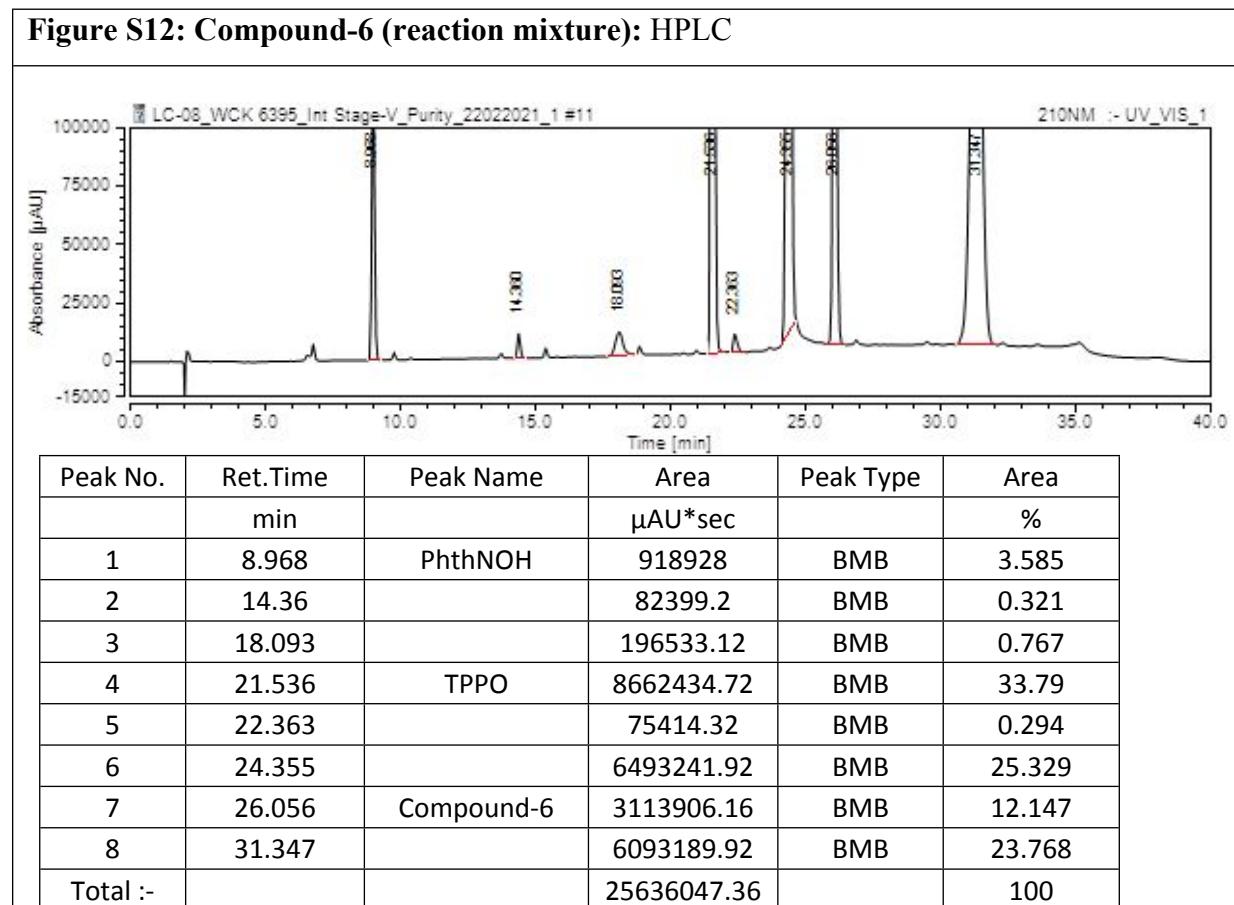


Figure S11: Compound-6: HPLC



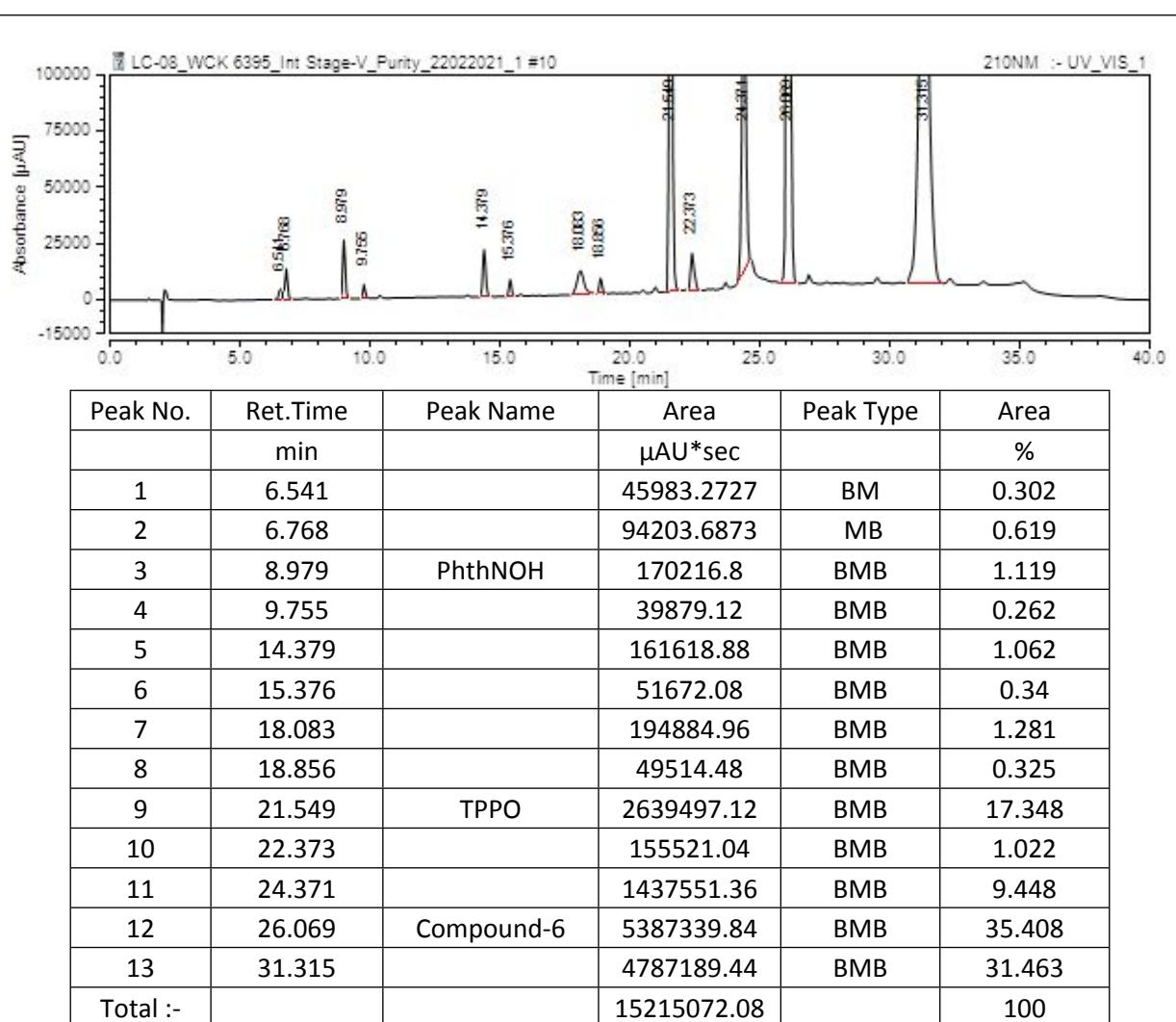
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Figure S12: Compound-6 (reaction mixture): HPLC



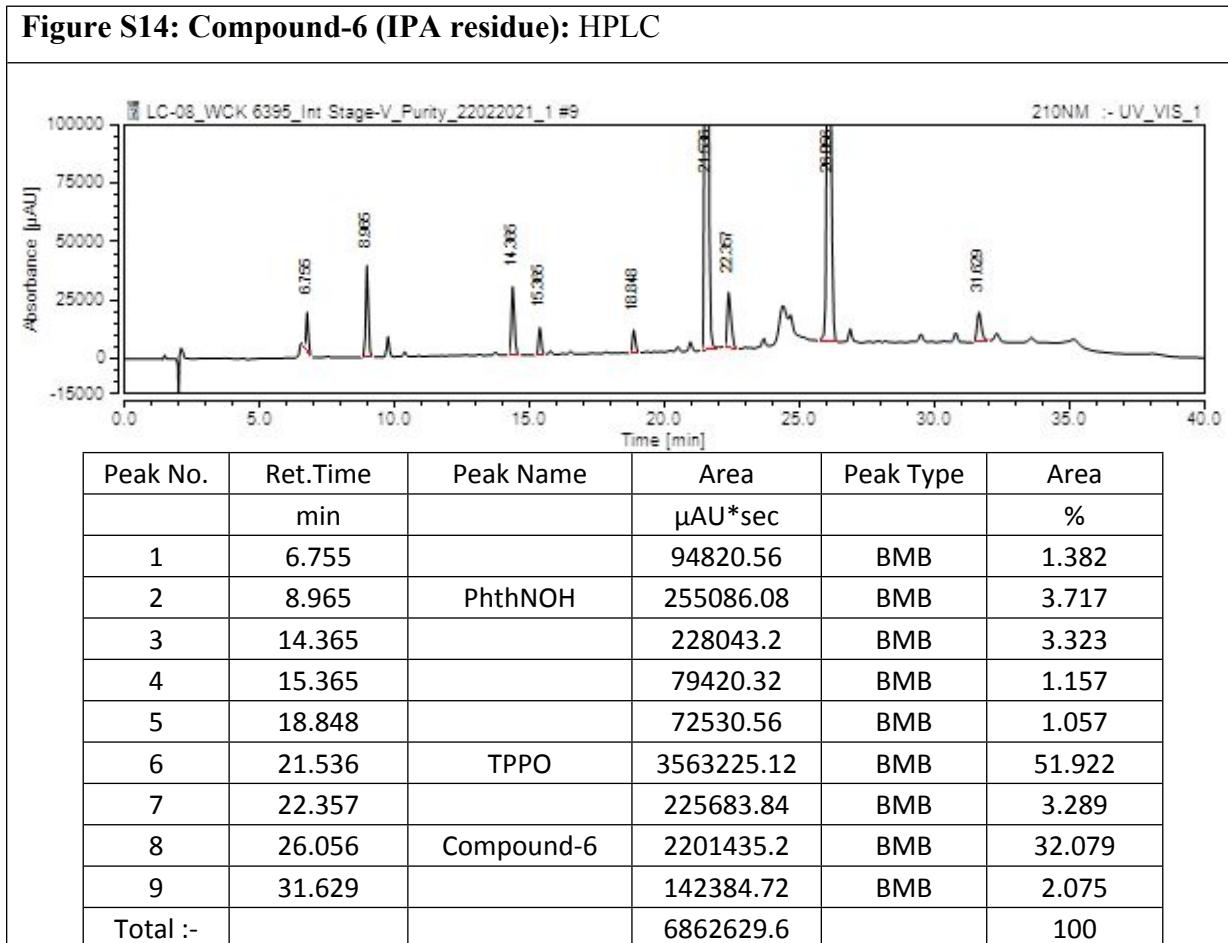
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Figure S13: Compound-6 (toluene residue): HPLC



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Figure S14: Compound-6 (IPA residue): HPLC



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Figure S15: Compound-7: ^1H NMR, CDCl_3 , 400 MHz

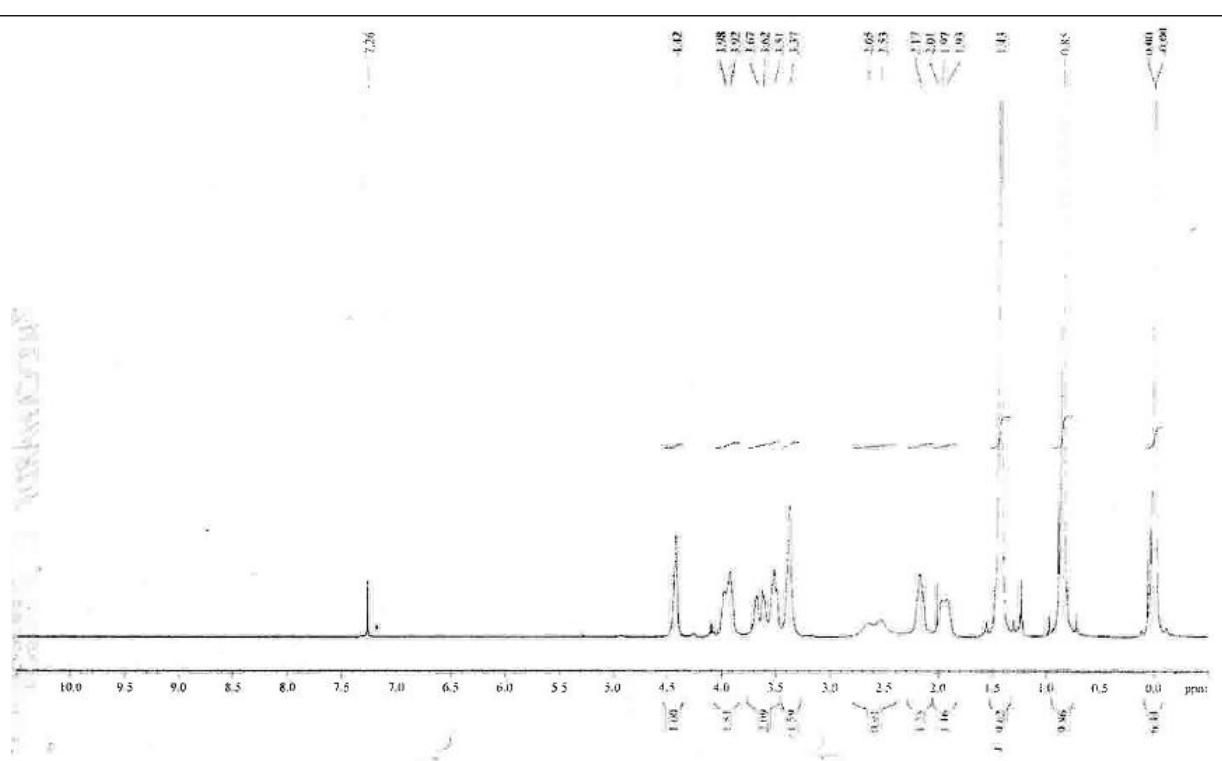
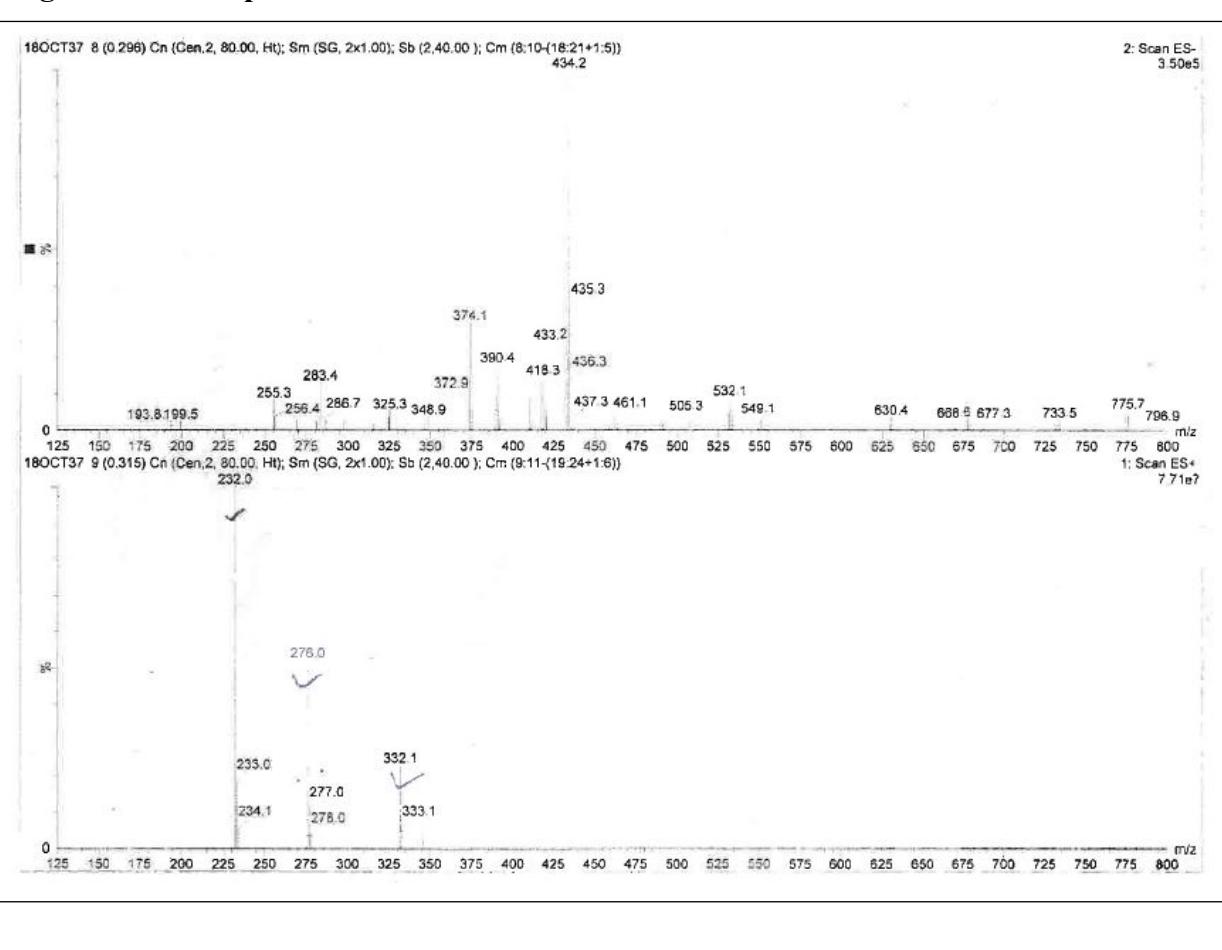


Figure S16: Compound-7: Mass



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Figure S17: Compound-8: ^1H NMR, CDCl_3 , 400 MHz

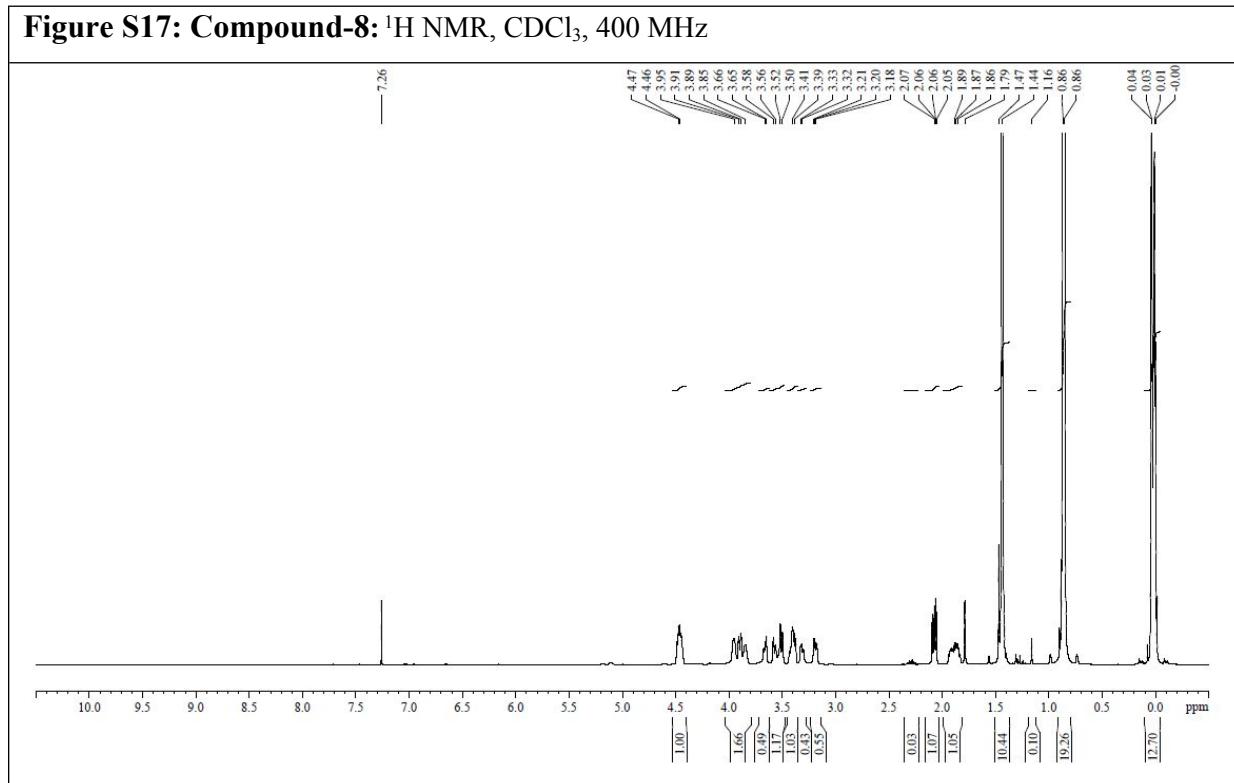
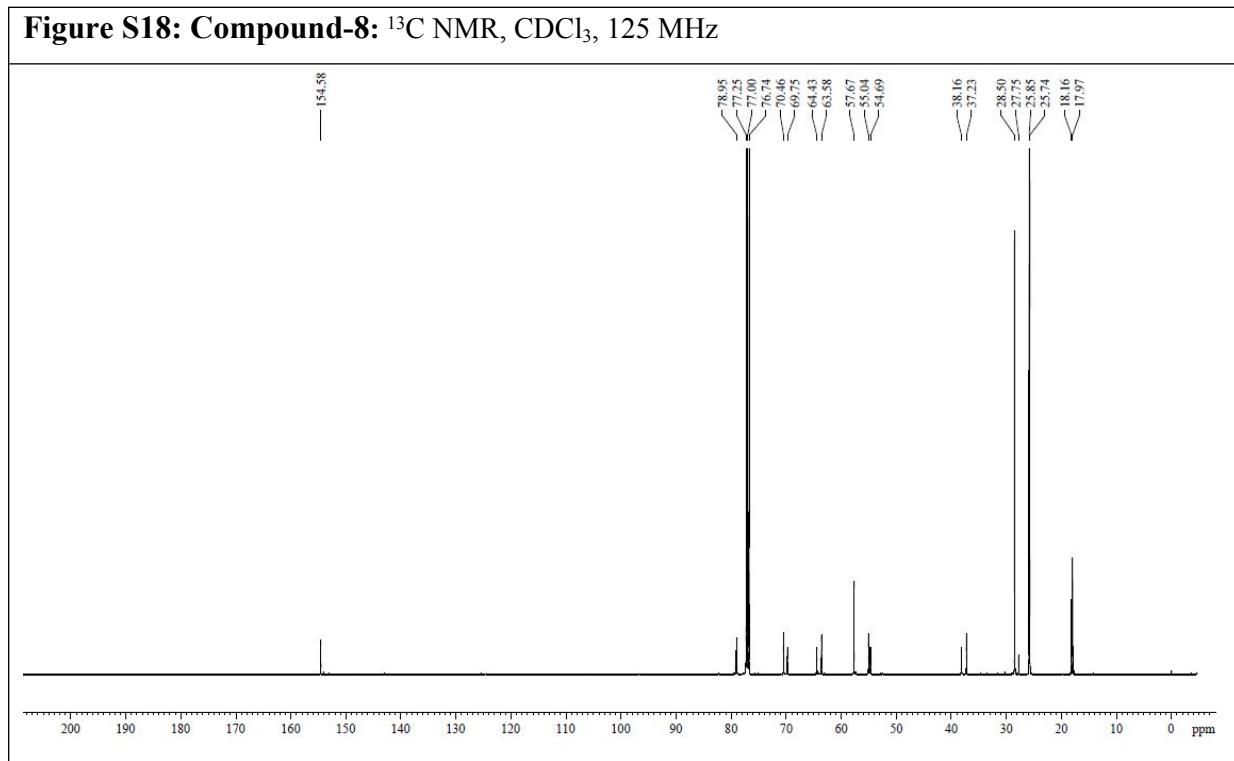


Figure S18: Compound-8: ^{13}C NMR, CDCl_3 , 125 MHz



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Figure S19: Compound-9: ^1H NMR, CDCl_3 , 400 MHz

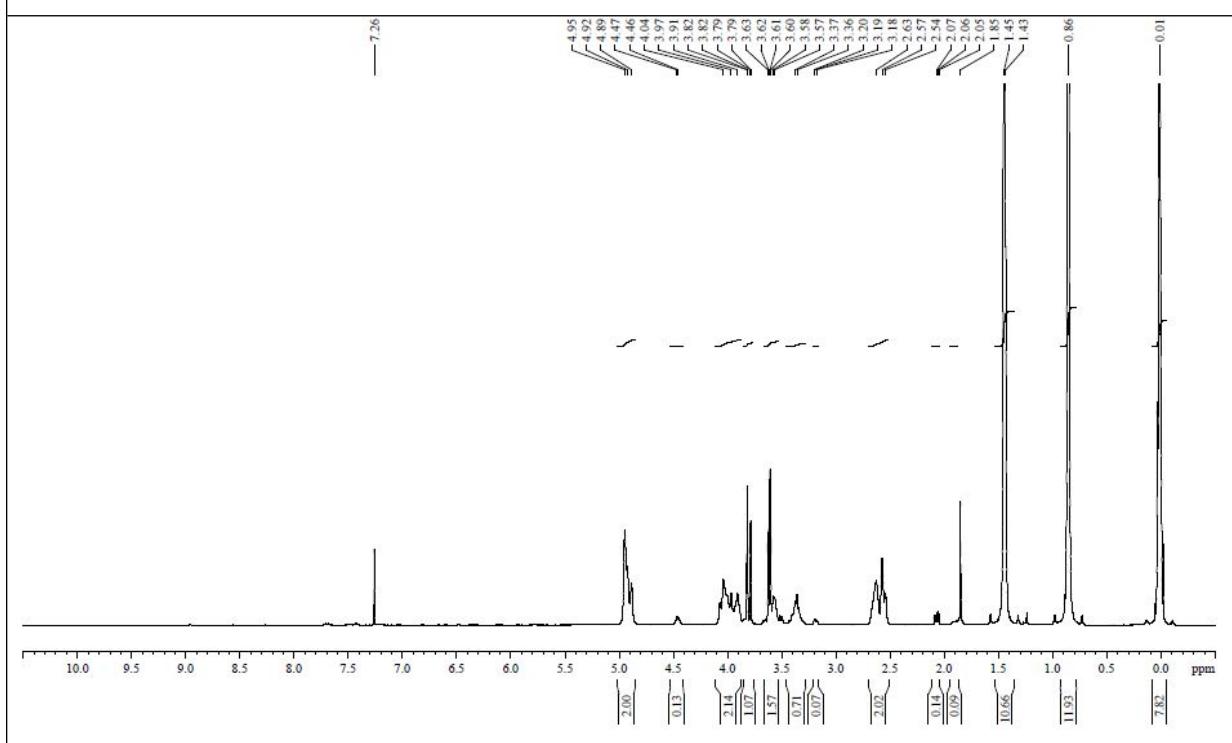
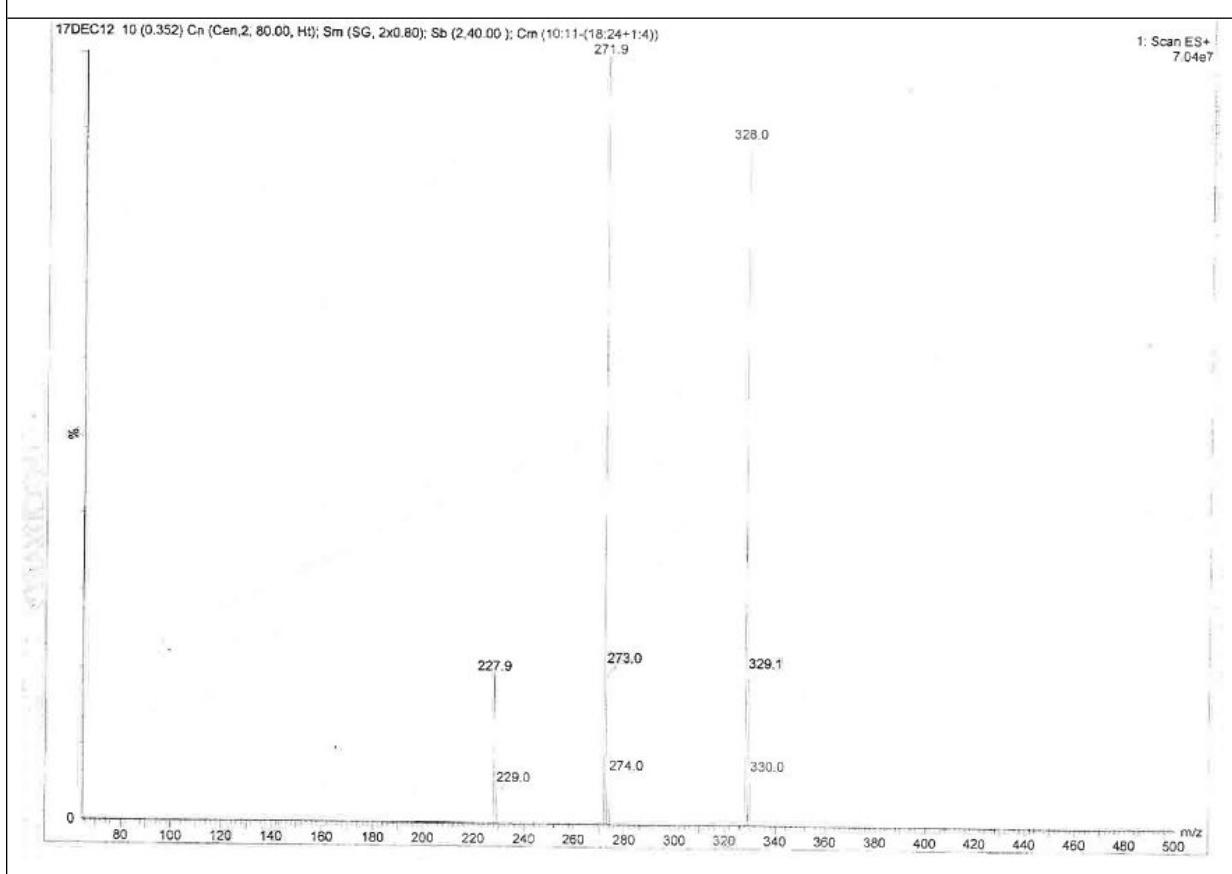
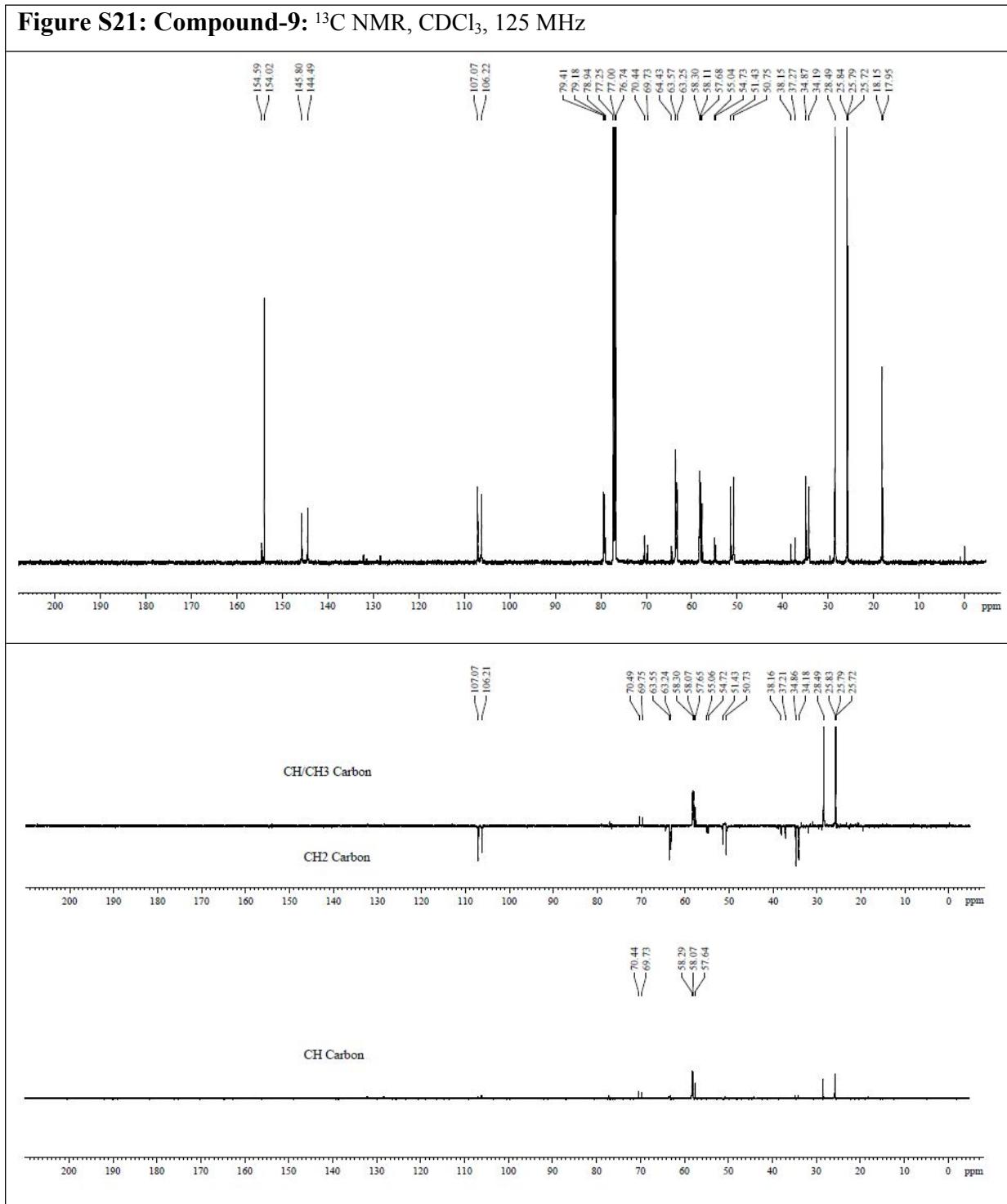


Figure S20: Compound-9: Mass



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Figure S21: Compound-9: ^{13}C NMR, CDCl_3 , 125 MHz



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Figure S22: Compound-10: ^1H NMR, CDCl_3 , 400 MHz

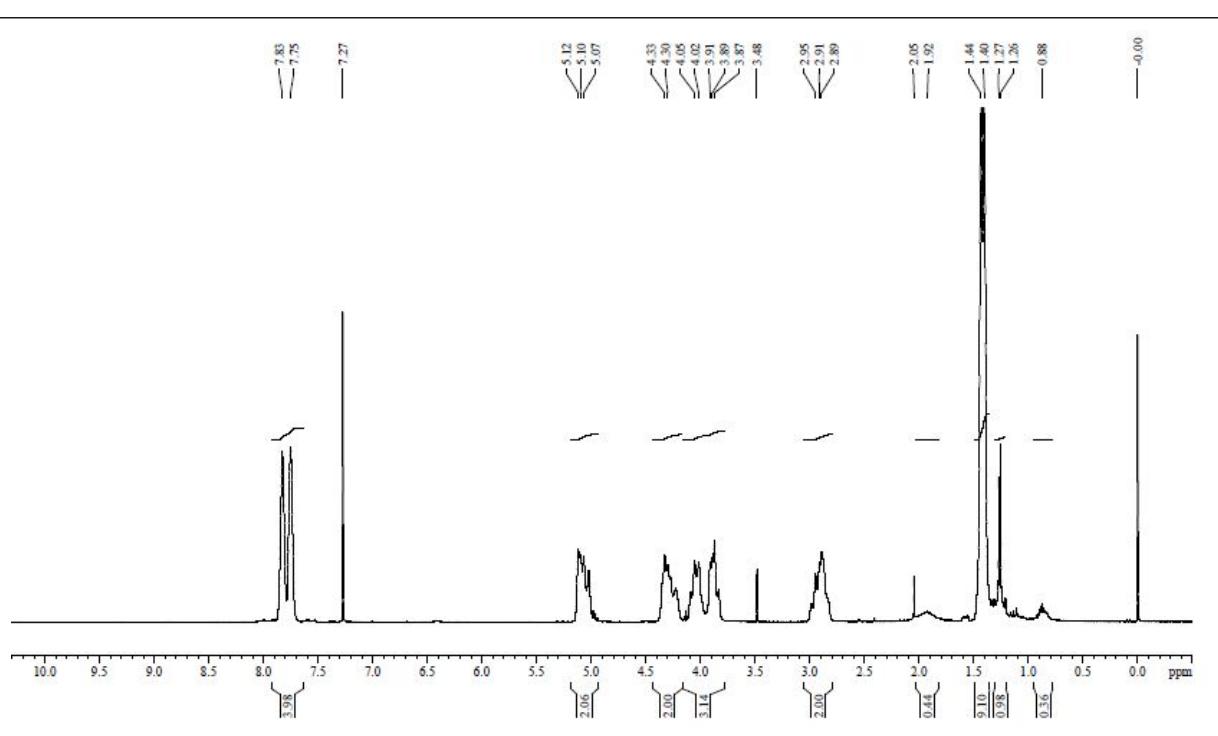
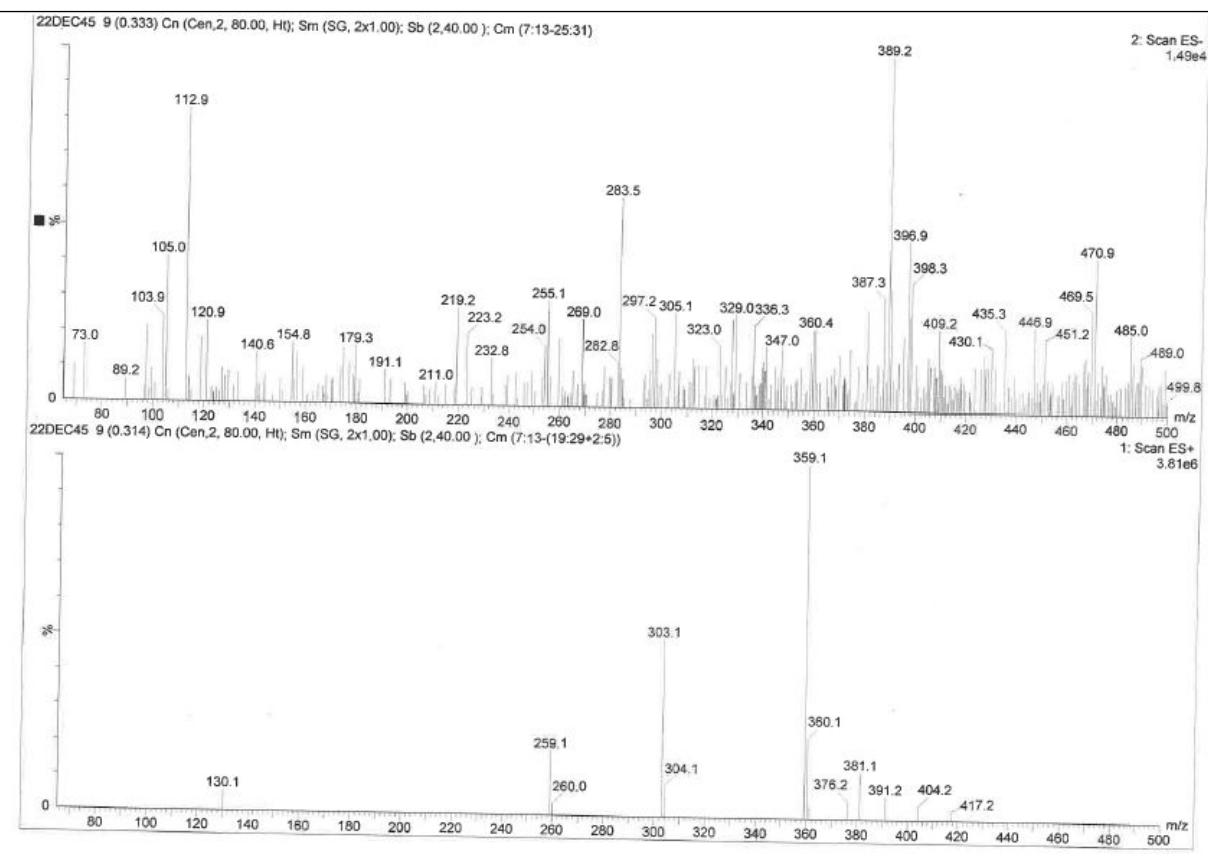


Figure S23: Compound-10: Mass



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Figure S24: Compound-10: ^{13}C NMR, CDCl_3 , 125 MHz

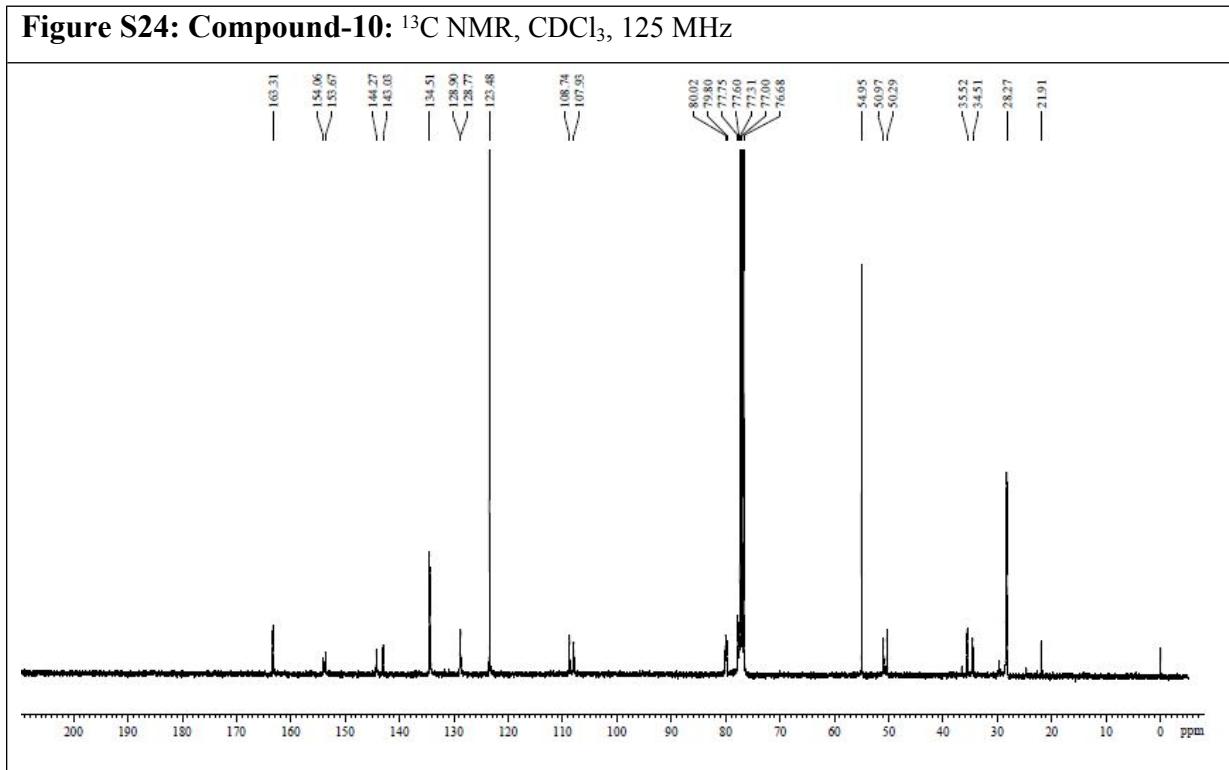
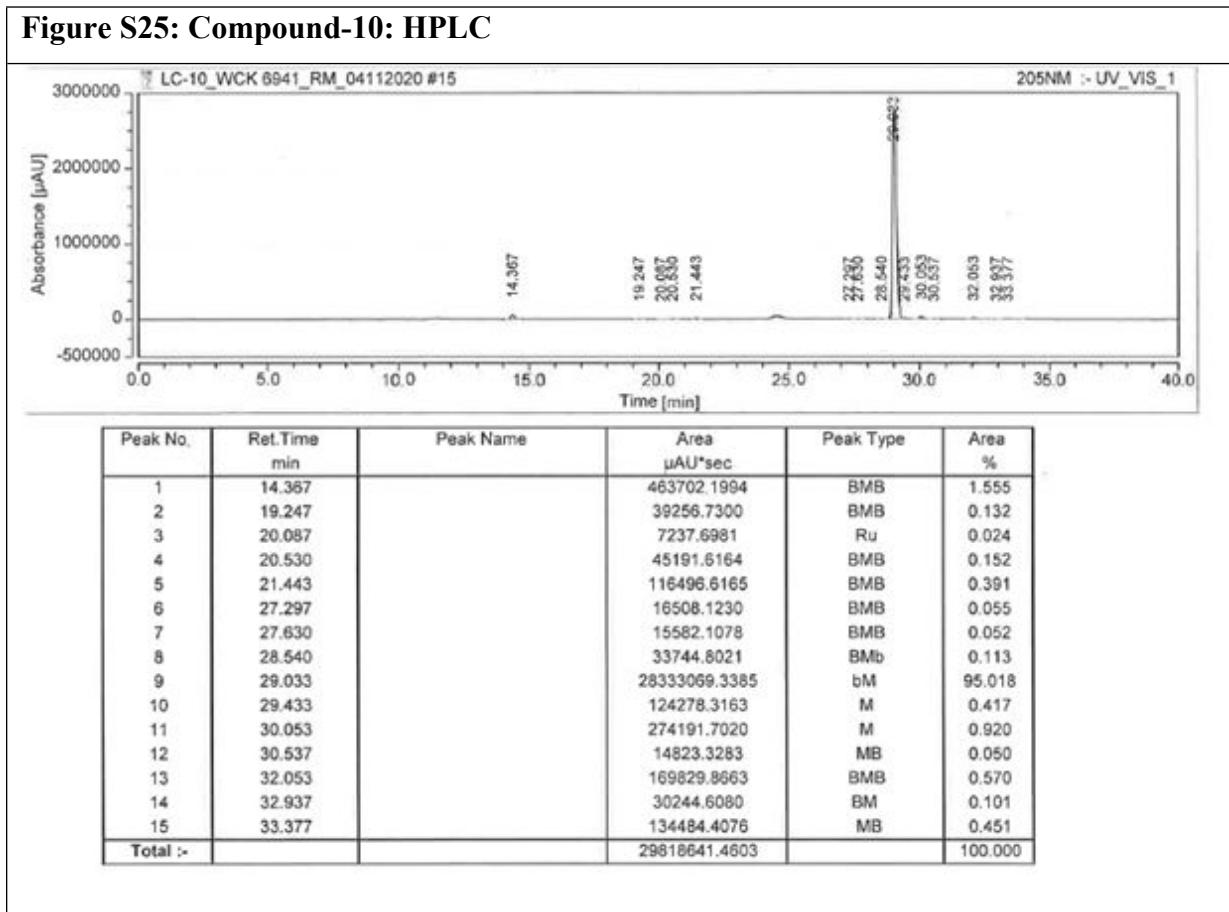


Figure S25: Compound-10: HPLC



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Figure S26: TPPO: Mass

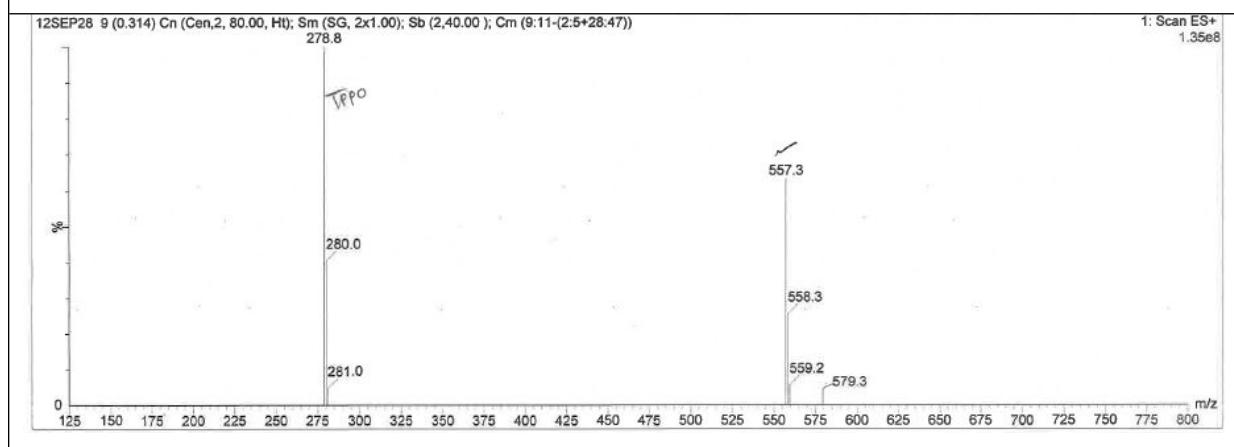
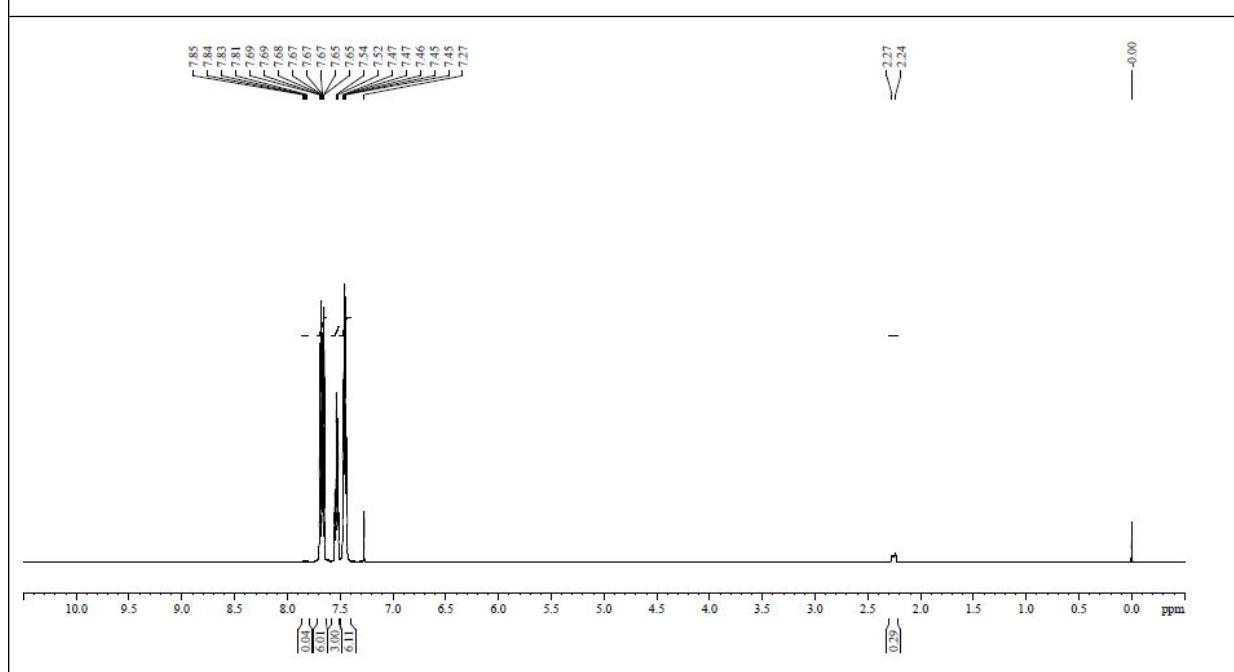


Figure S27: TPPO: NMR



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Figure S28: TPPO-H₂DIAD: NMR

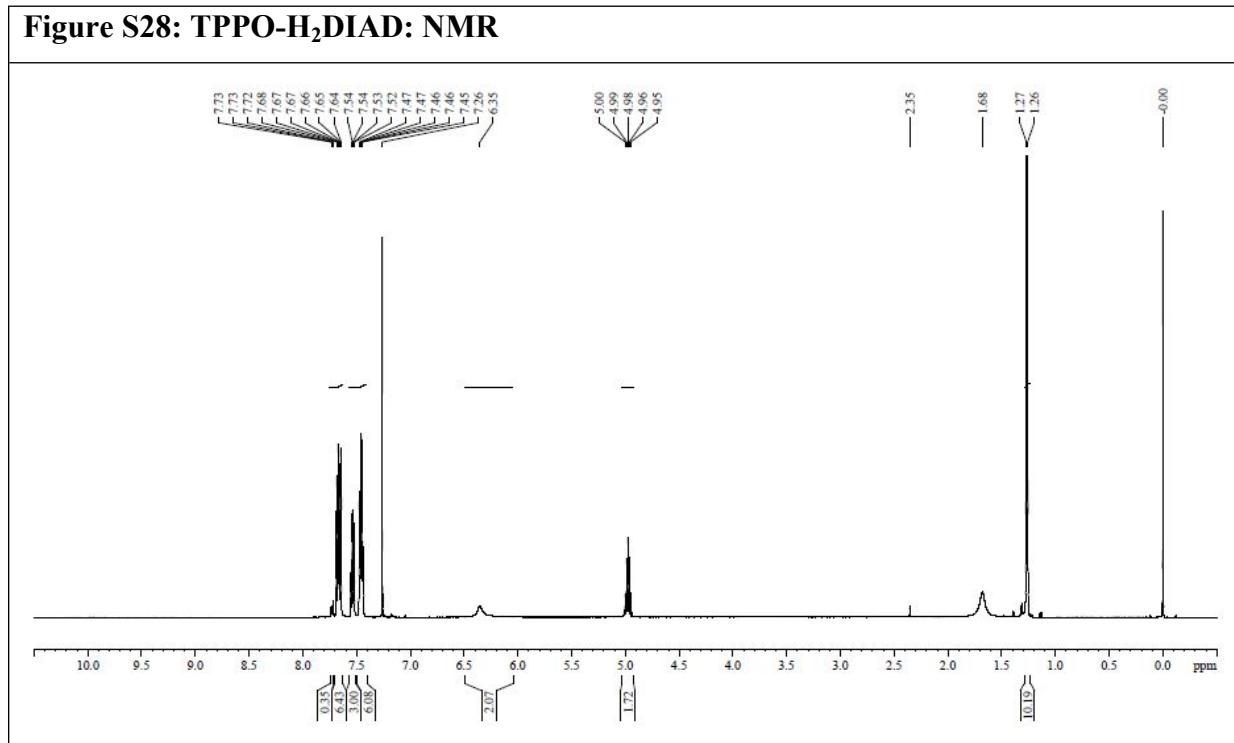
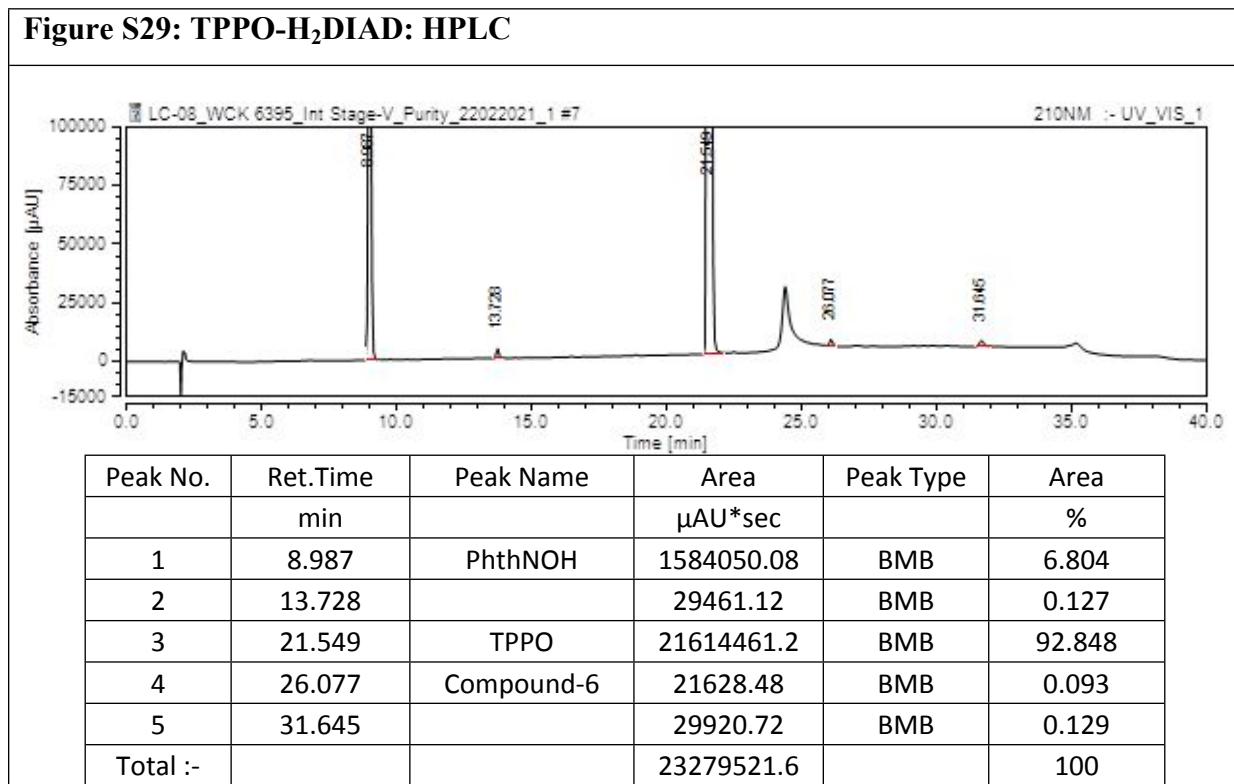


Figure S29: TPPO-H₂DIAD: HPLC



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Table S1

Compound-6									
Solubility at room temperature (25-30 °C)					Solubility at 0 °C				
Solvent	Weight (mg)	ml of solvent added	Solubility (mg/ml)	Observation	Solvent	Weight (mg)	ml of solvent added	Solubility (mg/ml)	Observation
2-Propanol	51.10	10.0	Insoluble	Insoluble particles	2-Propanol	51.60	10.0	Insoluble	Insoluble particles
Tetrahydrofuran	49.80	0.4	124.5	Clear Solution	Tetrahydrofuran	50.50	0.5	101.0	Clear Solution
Cyclohexane	49.91	10.0	Insoluble	Insoluble particles	Cyclohexane	50.90	10.0	Insoluble	Insoluble particles
Toluene	51.40	1.1	46.7	Clear Solution	Toluene	50.49	1.2	42.1	Clear Solution
TPPO-H ₂ DIAD									
2-Propanol	30.2	0.5	60.4	Clear Solution	2-Propanol	30.70	2.3	13.3	Clear Solution
Tetrahydrofuran	31.5	0.2	157.5	Clear Solution	Tetrahydrofuran	30.50	0.5	61.0	Clear Solution
Cyclohexane	30.1	10.0	Insoluble	Insoluble particles	Cyclohexane	32.00	10.0	Insoluble	Insoluble particles
Toluene	30.20	1.3	23.2	Clear Solution	Toluene	31.9	2.0	16.0	Clear Solution
TPPO									
2-Propanol	51.1	1.1	46.5	Clear Solution	2-Propanol	50.70	2.0	25.4	Clear Solution
Tetrahydrofuran	50.9	0.3	169.7	Clear Solution	Tetrahydrofuran	52.50	0.5	105.0	Clear Solution
Cyclohexane	50.4	10.0	Insoluble	Insoluble particles	Cyclohexane	50.80	10.0	Insoluble	Insoluble particles
Toluene	53.20	1.2	44.3	Clear Solution	Toluene	51.5	1.2	42.9	Clear Solution