Supporting Information for

A 2,2'-Bipyridyl Calcium Complex: Synthesis, Structure and Reactivity Studies

Lingfeng Wu, Zhenghui Wang, Yumiao Liu, Liang Chen,* Wenshan Ren*

College of Chemistry and Chemical Engineering, Southwest University, Chongqing 400715, China

rwsh603@swu.edu.cn (W. R.)

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EXPERIMENTAL SECTION

General Procedures. All reactions and manipulations were carried out under an atmosphere of dry dinitrogen with rigid exclusion of air and moisture using standard Schlenk or cannula techniques, or in a glove box. All organic solvents were freshly distilled from sodium benzophenone ketyl immediately prior to use. S_8 and azobenzene were purified by sublimation. {[CH_3C(N-2,6-$^iPr_2C_6H_3$)$CHC(CH_3)NCH_2CH_2N(CH_3)_2$]CaI(THF)}$_2$[1] and KC_8[2] were prepared according to literature methods. All other chemicals were purchased from Aldrich Chemical Co. and Energy Chemical Co. used as received unless otherwise noted. Infrared spectra were obtained from KBr pellets on an Avatar 360 Fourier transform spectrometer. Ultraviolet spectrum was performed on a Unico UV-4802H spectrophotometer. ^{1}H and $^{13}C\{^1H\}$ NMR spectra were recorded on a Bruker AV 600 spectrometer at 600 and 150 MHz, respectively. All chemical shifts were reported in δ units with reference to the residual protons of the deuterated solvents, which were internal standards, for proton and carbon chemical shifts. Electron paramagnetic resonance (EPR) spectroscopic measurement was performed on Bruker EMXplus console. Melting points were measured on an X-6 melting point apparatus and were uncorrected. Elemental analyses were performed on a Vario EL elemental analyzer.

Preparation of [CH_3C(N-2,6-$^iPr_2C_6H_3$)$CHC(CH_3)NCH_2CH_2N(CH_3)_2$]Ca(bipy)(THF) (1). KC_8 (0.035 g, 0.26 mmol) was added to a THF (5 mL) solution of {[CH_3C(N-2,6-$^iPr_2C_6H_3$)$CHC(CH_3)NCH_2CH_2N(CH_3)_2$]CaI(THF)}$_2$ (0.15 g, 0.13 mmol) and 2,2'-bipyridine

(0.041 g, 0.26 mmol). After stirring at 30 °C for 12 hours, the solution was filtered. The volume of the filtrate was reduced to 2 mL, dark red crystals of **1** were isolated at room temperature in the glovebox after addition of a few drops of n-hexane. Yield: 0.147 g (95%). M.p.: 124-128 °C. IR (KBr): *v* = 2958 (w), 2861 (w), 1620 (m), 1586 (w), 1546 (m), 1506 (w), 1458 (m), 1413 (s), 1336 (w), 1314 (w), 1258 (m), 1179 (m), 1145 (m), 1079 (m), 1021 (m), 934 (m), 800 (m) cm^{-1}. Anal. Calcd for $C_{35}H_{50}CaN_5O$: C, 70.43; H, 8.44; N, 11.73. Found: C, 70.30; H, 8.56; N, 11.54.

Preparation of $[CH_3C(N\text{-}2,6\text{-}^iPr_2C_6H_3)CHC(CH_3)NCH_2CH_2N(CH_3)_2]CaI(bipy)$ (2). A solution of I_2 (0.017 g, 0.065 mmol) in toluene (2 mL) was added dropwise to the toluene solution of $[CH_3C(N\text{-}2,6\text{-}^iPr_2C_6H_3)CHC(CH_3)NCH_2CH_2N(CH_3)_2]Ca(bipy)(THF)$ (0.078 g, 0.13 mmol) with stirring at room temperature. After 20 minutes, the solution was filtered. The solvent was removed, and the yellow crystals **2** were eventually obtained from a THF solution room temperature. Yield: 0.052 g (61%). M.p.: 154-156 °C. ^{1}H NMR (600 MHz, d_8-THF) δ = 9.25 (br, 2H, *H*-bipy), 8.37 (d, *J* = 7.2 Hz, 2H, *H*-bipy), 7.94 (br, 2H, *H*-bipy), 7.43 (br, 2H, *H*-bipy), 6.87 (br, 2H, *H*-Ph), 6.82 (m, 1H, *H*-Ph), 4.57 (s, 1H, MeC(N)C*H*), 3.58 (thf), 3.37 (t, *J* = 5.4 Hz, 2H, NCH_2), 2.99 (br, 2H, NCH_2), 2.70 (br, 2H, ArC*H*Me$_2$), 1.93-1.82 (m, 9H, N*Me*$_2$ and C*Me*), 1.74 (thf), 1.46 (s, 3H, C*Me*), 1.11-0.86 (m, 12H, ArCH*Me*$_2$). ^{13}C NMR (151 MHz, d_8-THF) δ = 167.5 (imine-*C*), 165.7 (imine-*C*), 156.0 (*C*-bipy), 152.5 (*C*-bipy), 149.9 (*C*-Ph), 143.2 (*C*-Ph), 139.5 (*C*-bipy), 126.0, 124.5, 124.3, 122.7 (*C*-bipy and *C*-Ph), 95.1 (MeC(N)*C*H), 68.0 (thf), 60.7 (N*C*H$_2$) , 49.0 (N*C*H$_2$), 46.4 (N*Me*$_2$), 29.2 (Ar*C*HMe$_2$), 27.0 (ArCH*Me*$_2$),

25.9 (thf), 25.5 (C*Me*), 23.5 (C*Me*). IR (KBr): *v* = 2961 (w), 2861 (w), 1619 (m), 1537 (m), 1513 (m), 1465 (m), 1412 (m), 1383 (m), 1336 (w), 1310 (w), 1259 (m), 1179 (s), 1146 (s), 1078 (s), 1021 (m), 800 (m) cm^{-1}. Anal. Calcd for $C_{31}H_{42}CaN_5I$: C, 57.13; H, 6.50; N, 10.75. Found: C, 57.02; H, 6.59; N, 10.59.

Reduction of compound 2. To a THF (5 mL) solution of **2** (0.085 g, 0.13 mmol) was added KC_8 (0.018 g, 0.13 mmol). After stirring at 30 °C for 12 hours, the solution was filtered. The volume of the filtrate was reduced to 2 mL, dark red crystals of **1** were isolated at room temperature in the glovebox after addition of a few drops of n-hexane. Yield: 0.075 g (96%).

Preparation of [CH_3C(N-2,6-$^i Pr_2C_6H_3$)$CHC(CH_3)NCH_2CH_2N(CH_3)_2$]$CaSCH_2Ph$(bipy) (3). A solutoin of $PhCH_2SSCH_2Ph$ (0.015 g, 0.06 mmol) in THF (1 mL) was added to the solution of **1** (0.072 g, 0.12 mmol) in THF (2 mL) with stirring at room temperature. After 2 hours, the solution was filtered. The volume of the filtrate was reduced to 2 mL, yellow crystals of **3** were isolated at room temperature in the glovebox after addition of a few drops of DME. Yield: 0.052 g (67%). M.p.: 156-158 °C. ^{1}H NMR (600 MHz, d_8-THF) δ = 9.15 (br, 2H, *H*-bipy), 8.36 (d, *J* = 7.2 Hz, 2H, *H*-bipy), 7.92 (br, 2H, *H*-bipy), 7.32 (br, 2H, *H*-bipy), 7.10 (d, *J* = 7.2 Hz, 2H, *H*-Ph), 7.00-6.97 (m, 4H, *H*-Ph), 6.91-6.85 (m, 2H, *H*-Ph), 4.62 (s, 1H, MeC(N)C*H*), 3.58 (thf), 3.36 (m, 2H, NCH_2), 3.07-2.87 (m, 4H, NCH_2 and CH_2Ph), 2.60 (br, 2H, Ar*CH*Me_2), 1.97 (s, 3H, C*Me*), 1.90 (s, 6H, NMe_2), 1.73 (thf), 1.54 (s, 3H, C*Me*), 1.14-0.89 (m, 12H, ArCHMe_2). ^{13}C NMR (151 MHz, d_8-THF) δ = 167.6 (imine-*C*), 165.2 (imine-*C*), 156.5 (*C*-bipy), 153.0 (*C*-bipy),

151.2 (*C*-bipy), 150.2(*C*-Ph), 143.7 (*C*-Ph), 139.6 (*C*-bipy),130.1, 128.4, 125.9, 125.2, 124.7, 122.6 (*C*-bipy and *C*-Ph), 95.1 (MeC(N)*C*H), 68.1 (thf), 61.0 (N*C*H_2), 49.2 (N*C*H_2), 46.2 (N*Me*$_2$), 31.8 (*C*H_2Ph), 29.4 (Ar*C*HMe$_2$), 26.1 (thf), 25.6 (ArCH*Me*$_2$), 25.5 (C*Me*), 23.6(C*Me*). IR (KBr): *v* = 2951 (w), 2864 (w), 1597 (w), 1543 (m), 1512 (m), 1465 (m), 1411 (m), 1334 (m), 1234 (m), 1180 (m), 1095 (m), 1056 (m), 1007 (m), 933 (m) cm^{-1}. Anal. Calcd for $C_{38}H_{49}CaN_5S$: C, 70.44; H, 7.62; N, 10.81. Found: C, 70.31; H, 7.71; N, 10.70.

Preparation of [CH$_3$C(N-2,6-^{i}Pr$_2$C$_6$H$_3$)CHC(CH$_3$)NCH$_2$CH$_2$N(CH$_3$)$_2$]CaSeCH$_2$Ph(bipy) (4). This compound was prepared as yellow crystals from the reaction of **1** (0.072 g, 0.12 mmol) and PhCH$_2$SeSeCH$_2$Ph (0.021 g, 0.06 mmol) in THF (3 mL) at room temperature and recrystallization from a THF/DME/n-haxane solution by a procedure similar to that described in the synthesis of **3**. Yield: 0.046 g (55%).. M.p.: 134-136 ^{o}C. ^{1}H NMR (600 MHz, d$_8$-THF) δ = 9.17 (br, 2H, *H*-bipy), 8.36 (d, *J* = 7.8 Hz, 2H, *H*-bipy), 7.93 (br, 2H, *H*-bipy), 7.33 (br, 2H, *H*-bipy), 7.00 (m, 4H, *H*-Ph), 6.93 (t, *J* = 7.8 Hz, 2H, *H*-Ph), 6.90 (t, *J* = 7.8 Hz, 1H, *H*-Ph), 6.82 (t, *J* = 7.8 Hz, 1H, *H*-Ph), 4.63 (s, 1H, MeC(N)C*H*), 3.58 (thf), 3.37 (t, *J* = 6.6 Hz, 2H, NC*H*$_2$), 3.07 (br, 2H, C*H*$_2$Ph), 2.89 (m, 2H, NC*H*$_2$), 2.60 (br, 2H, ArC*H*Me$_2$), 1.97 (s, 3H, C*Me*), 1.87 (s, 6H, N*Me*$_2$), 1.73 (thf), 1.54 (s, 3H, C*Me*), 1.14-0.89 (m, 12H, ArCH*Me*$_2$). ^{13}C NMR (151 MHz, d$_8$-THF) δ = 167.4 (imine-*C*), 165.1 (imine-*C*), 155.8 (*C*-bipy), 153.3 (*C*-bipy), 151.1 (*C*-Ph), 150.0 (*C*-Ph),143.2 (*C*-Ph), 139.6 (*C*-bipy),129.9, 128.1, 125.8, 124.6, 124.5, 122.5 (*C*-bipy and *C*-Ph), 94.6 (MeC(N)*C*H), 67.9 (thf), 60.7 (N*C*H_2), 48.9 (N*C*H_2), 46.0 (N*Me*$_2$), 29.2

(Ar*C*HMe$_2$ and *C*H$_2$Ph), 25.8 (thf), 25.4 (ArCH*Me*$_2$), 25.2 (C*Me*), 23.3(C*Me*). IR (KBr): *v* = 2958 (w), 2850 (w), 1535 (m), 1512(m), 1458(m), 1411(m), 1334(m), 1234(m), 1165(m), 1056(m), 1002(s), 933(m) cm^{-1}. Anal. Calcd for $C_{38}H_{49}CaN_5Se$: C, 65.68; H, 7.11; N, 10.08. Found: C, 65.83; H, 7.24; N, 9.98.

Preparation of [CH$_3$C(N-2,6-^{i}Pr$_2$C$_6$H$_3$)CHC(CH$_3$)NCH$_2$CH$_2$N(CH$_3$)$_2$]Ca[O-(9-C$_{13}$H$_8$)](bipy)·2THF (5·2THF). A solution of 9-fluorenone (0.024 g, 0.13 mmol) in THF (2 mL) was added dropwise to the THF solution of **1** (0.078 g, 0.13 mmol) with stirring at room temperature. After 20 minutes, the solution was filtered. The volume of the filtrate was reduced to 2 mL, and the compound **5** crystallized at room temperature as dark crystals. Yield: 0.094 g (85%). M.p.: 249-253 ºC. IR (KBr): *v* = 2958 (w), 2862 (w), 1618 (m), 1586 (m),1541 (m), 1508 (m), 1456 (m), 1413 (m), 1337 (w), 1316 (w), 1258 (m), 1174 (m), 1088 (s), 1021 (s), 916 (m), 804 (m) cm^{-1}. Anal. Calcd for $C_{52}H_{66}CaN_5O_3$: C, 73.55; H, 7.83; N, 8.25. Found: C, 73.69; H, 7.75; N, 8.37.

Preparation of {[S$_2$CC(CMe(NAr))C(Me)NCH$_2$CH$_2$NMe$_2$]Ca(DME)}$_2$ (6). CS_2 (0.0152 g, 0.20 mmol) was added to the solution of **5** (0.141 g, 0.20 mmol) in THF (3 mL) with stirring at room temperature. After 20 minutes, the solvent was removed. The residue was extracted with DME (3 mL × 3) and filtered. Concentration to approximately 2 mL and storage at room temperature gave **6** as light yellow crystals, which were isolated by filtration. Yield: 0.070 g (65%). This compound was

characterized by ^{1}H NMR spectroscopy and single crystal X-ray diffraction studies, which are identical to the previous report.[1]

Preparation of {[(9-CH_2=$CHCH_2$-$C_{13}H_8$-9)-O]CaBr(THF)(bipy)}$_2$ (7). CH_2=$CHCH_2Br$ (0.024 g, 0.20 mmol) was added to the solution of **5** (0.141 g, 0.20 mmol) in DME/THF (3 mL) with stirring at room temperature. After 1 hour, the solution was filtered. The volume of the filtrate was reduced to 2 mL, light yellow crystals of **7** were isolated at room temperature in the glovebox after addition of a few drops of THF. Yield: 0.047 g (41 %). M.p.: 188-190 ^{o}C (decompose). IR (KBr): *v* = 3032 (w), 2976 (w), 2872 (w), 1632 (w), 1597 (s), 1576 (m), 1472 (m), 1433(m), 1386 (m), 1316 (w), 1299 (w), 1256 (w), 1200 (w), 1174 (w), 1148 (m), 1104 (m), 1065 (s), 1040 (m), 1009 (m), 923 (m) cm^{-1}. Anal. Calcd for $C_{60}H_{58}Br_2Ca_2N_4O_4$: C, 63.27; H, 5.13; N, 4.92. Found: C, 63.08; H, 5.02; N, 4.81. This compound was insoluble in deuterated solvents such as pyridine, THF, benzene and toluene, which made the characterization by NMR spectroscopy infeasible.

Preparation of {[(9-$C_6H_5CH_2$-$C_{13}H_8$-9)-O]CaBr[O-(9-$C_{13}H_8$)](bipy)}$_2$ (8). $PhCH_2Br$ (0.034 g, 0.20 mmol) was added to the solution of **5** (0.141 g, 0.20 mmol) in DME (3 mL) with stirring at room temperature. After 1 hour, the solution was filtered. The volume of the filtrate was reduced to 2 mL, yellow crystals of **8** were isolated at room temperature in the glovebox after addition of a few drops of benzene. Yield: 0.027 g (34%). M.p.: 228-230 ^{o}C (decompose). IR (KBr): *v* = 3032 (w), 2911 (w), 2864 (w), 1714 (s), 1610 (m), 1597 (s), 1576 (m), 1472 (m), 1450 (m), 1433(m), 1381 (m),

1299 (m), 1187 (m), 1148 (m), 1109 (m), 1065 (s), 1009 (m), 923 (m) cm^{-1}. Anal. Calcd for $C_{86}H_{62}Br_2Ca_2N_4O_4$: C, 70.97; H, 4.29; N, 3.85. Found: C, 70.81; H, 4.18; N, 3.72. This compound was insoluble in deuterated solvents such as pyridine, THF, benzene and toluene, which made the characterization by NMR spectroscopy infeasible.

***X*-ray Crystallography.** Single-crystal X-ray diffraction measurements were carried out on an Agilent SuperNova EosS2 diffractometer using graphite monochromated Cu Kα radiation (λ = 1.54184 Å) or Mo Kα radiation (λ = 0.71073 Å). The crystals were kept at 180 (10) K during data collection. The structures were solved by the Superflip[3] or ShelXT[4] structure solution program in Olex2[5] and refined using Full-matrix Least Squares based on F^2 with program SHELXL-2018[6] within Olex2. Disorder was modelled using standard crystallographic methods including constraints and restraints where necessary. Crystal data and experimental data for **1-5**, **7**and **8** are summarized in Table S1. For compound **4**, the solvent molecule was disordered and could not be modeled properly; thus, the OLX2 solvent mask was used to calculate the solvent disorder area and remove its contribution to the overall intensity data.

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ORTEP DIAGRAM

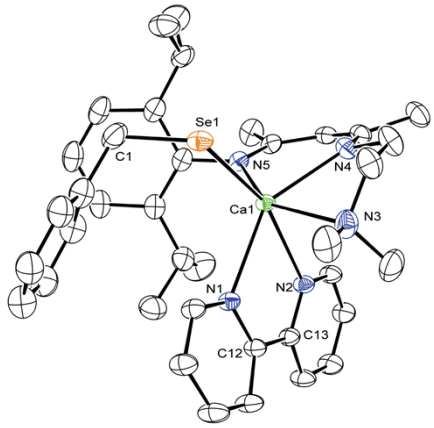

Fig. S1 ORTEP representation of **4**. Displacement ellipsolids are set to 30% probability. The hydrogen atoms are omitted for clarity. Selected distances (Å) and angles (°): Ca(1)-Se(1) 2.9324(5), Ca(1)-N(1) 2.543(2), Ca(1)-N(2) 2.5293(19), Ca(1)-N(3) 2.595(2), Ca(1)-N(4) 2.390(2), Ca(1)-N(5) 2.425(2), C(12)-C(13) 1.489(3), N(2)-Ca(1)-N(1) 64.37(6), N(1)-C(12)-C(13)-N(2) 17.23.

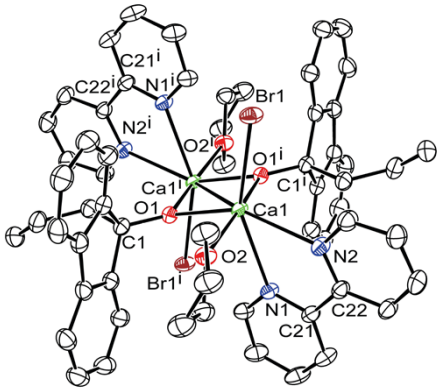

Fig. S2 ORTEP representation of **7**. Displacement ellipsolids are set to 30% probability. The hydrogen atoms are omitted for clarity. Selected distances (Å) and angles (°): Ca(1)-Br(1) 2.8861(5), Ca(1)-O(1) 2.2707(17), Ca(1i)-O(1) 2.3315(17), Ca(1)-O(2) 2.4317(19), Ca(1)-N(1) 2.512(2), Ca(1)-N(2) 2.626(2), O(1)-C(1) 1.402(3), C(21)-C(22) 1.486(4), N(2)-Ca(1)-N(1) 62.70(7), Ca···Ca 3.5756(9), N(1)-C(38)-C(39)-N(2) 2.17.

Table S1. Crystal data and experimental parameters for compounds **1**-**5, 7** and **8**

Compound	**1**	**2**	**3**	**4**	**5·2THF**	**7**	**8**
Formula	$C_{35}H_{50}CaN_5O$	$C_{31}H_{42}CaIN_5$	$C_{38}H_{49}CaN_5S$	$C_{38}H_{49}CaN_5Se$	$C_{52}H_{66}CaN_5O_3$	$C_{60}H_{58}Br_2Ca_2N_4O_4$	$C_{86}H_{62}Br_2Ca_2N_4O_4$
Fw	596.88	651.67	647.96	694.86	849.17	1139.08	1455.37
Crystal system	triclinic	triclinic	monoclinic	orthorhombic	monoclinic	monoclinic	triclinic
Space group	P-1	P-1	$P2_1$	Pbcn	$P2_1/c$	$P2_1/n$	P-1
a/Å	10.2949(7)	9.9398(3)	10.59500(10)	22.5884(3)	13.30148(10)	12.2707(2)	11.1141(4)
b/Å	10.3859(8)	10.1072(4)	17.4079(2)	18.63906(18)	20.12994(16)	12.5295(2)	12.0745(3)
c/Å	17.1736(12)	17.2990(6)	10.66230(10)	19.03955(18)	18.18427(15)	17.6850(2)	14.6995(5)
α/°	78.478(6)	89.334(3)	90	90	90	90	78.371(2)
β/°	81.896(6)	74.032(3)	109.7740(10)	90	95.7924(7)	95.4230(10)	70.616(3)
γ/°	67.135(7)	70.382(3)	90	90	90	90	68.461(3)
Volume/Å^3	1653.7(2)	1567.60(9)	1850.56(3)	8016.14(14)	4844.12(7)	2706.82(7)	1723.70(10)
Z	2	2	2	8	4	2	1
ρ_{calc}g/cm^3	1.195	1.381	1.163	1.152	1.164	1.398	1.402
μ/mm^{-1}	1.892	1.212	2.225	2.606	1.469	3.956	3.242
F(000)	642.0	672.0	696.0	2928.0	1828.0	1176	748.0
Crystal size/mm^3	0.2 × 0.2 × 0.15	0.2 × 0.2× 0.2	0.2 × 0.2 × 0.05	0.2 × 0.05 × 0.05	0.2 × 0.1× 0.03	0.1 × 0.05 × 0.05	0.15 × 0.1 × 0.05
Radiation	CuKα	MoKα	CuKα	CuKα	CuKα	CuKα	CuKα
2θ range (deg)	9.346 to 145.976	6.814 to 58.596	8.814 to 134.13	7.706 to 143.764	7.996 to 143.712	8.41 to 143.526	7.904 to 143.578
Reflections collected	23974	27744	19450	17326	36395	15284	19188
Independent reflections	6473 [R_{int}= 0.0459]	7607 [R_{int}= 0.0336]	6373 [R_{int}= 0.0275]	7694[R_{int}= 0.0187]	9379[R_{int}= 0.0278]	5241[R_{int}= 0.0287]	6652[R_{int}= 0.0285]
Data/restr/paras	6473/6/408	7607/0/359	6373/86/433	7694/36/410	9379/87/585	5241/40/343	6652/0/442
GOF	1.020	1.153	1.044	1.037	1.031	1.032	1.022
R1/wR2 [$I >= 2\sigma(I)$]	0.0538 / 0.1370	0.0358 / 0.0796	0.0512 / 0.1328	0.0436 / 0.1151	0.0546 / 0.1520	0.0382 / 0.1021	0.0290 / 0.0754
R1/wR2 (all data)	0.0664 / 0.1460	0.0422 / 0.0821	0.0521 / 0.1340	0.0506 / 0.1203	0.0574 / 0.1551	0.0448 / 0.1075	0.0316 / 0.0778

Largest diff. peak/hole / e $Å^{-3}$	0.85/-0.56	0.91/-0.53	0.56/-0.48	0.87/-0.81	0.91/-0.58	0.67/-0.42	0.44/-0.23
CCDC	2236852	2236853	2236854	2236855	2236857	2249790	2236856

EPR SPECTRUM

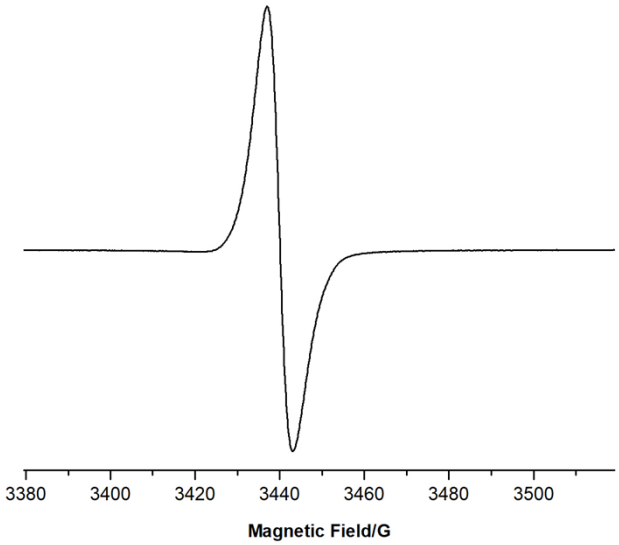

Fig. S3 Experimental 295 K EPR spectrum of complex **1** in THF.

UV-vis ABSORPTION SPECTRA

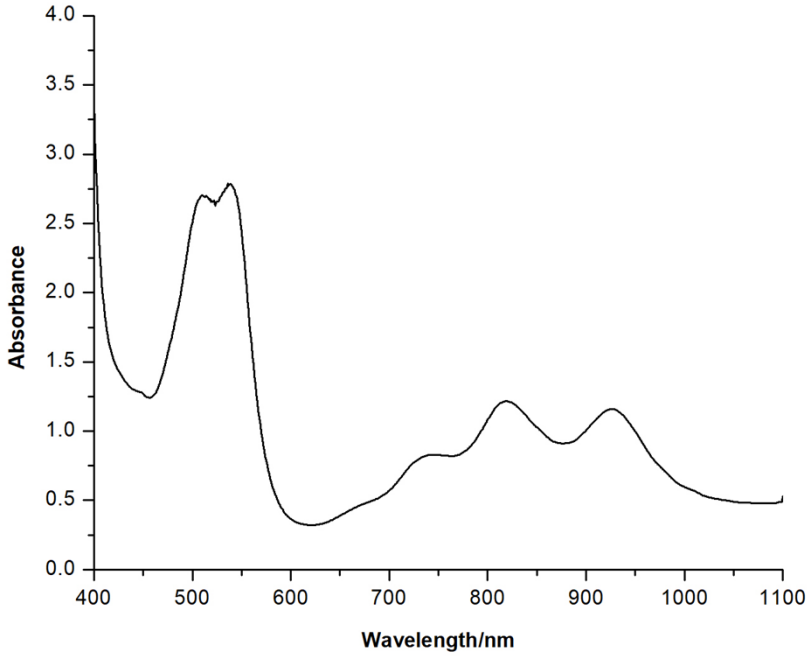

Fig. S4 UV-Vis spectrum of compound **1** (concentration is 8.5×10^{-4} M in THF).

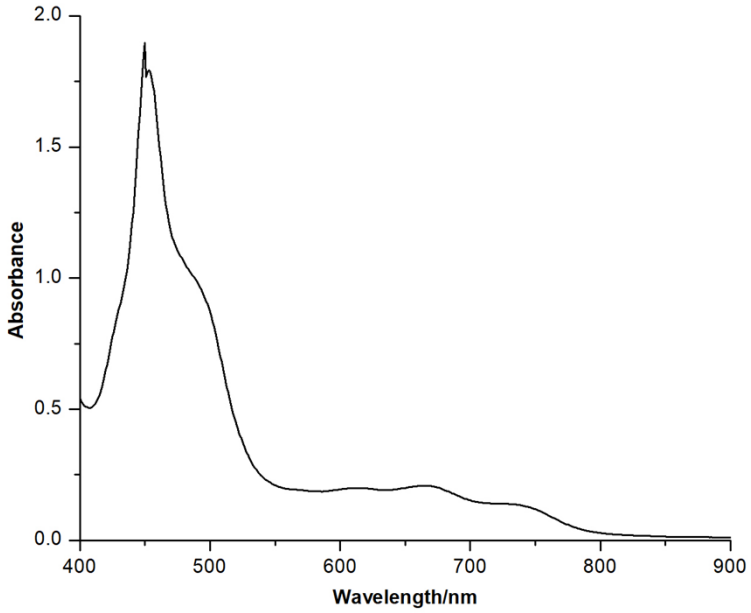

Fig. S5 UV-Vis spectrum of compound **5** (concentration is 8.5×10^{-4} M in THF).

NMR SPECTRA

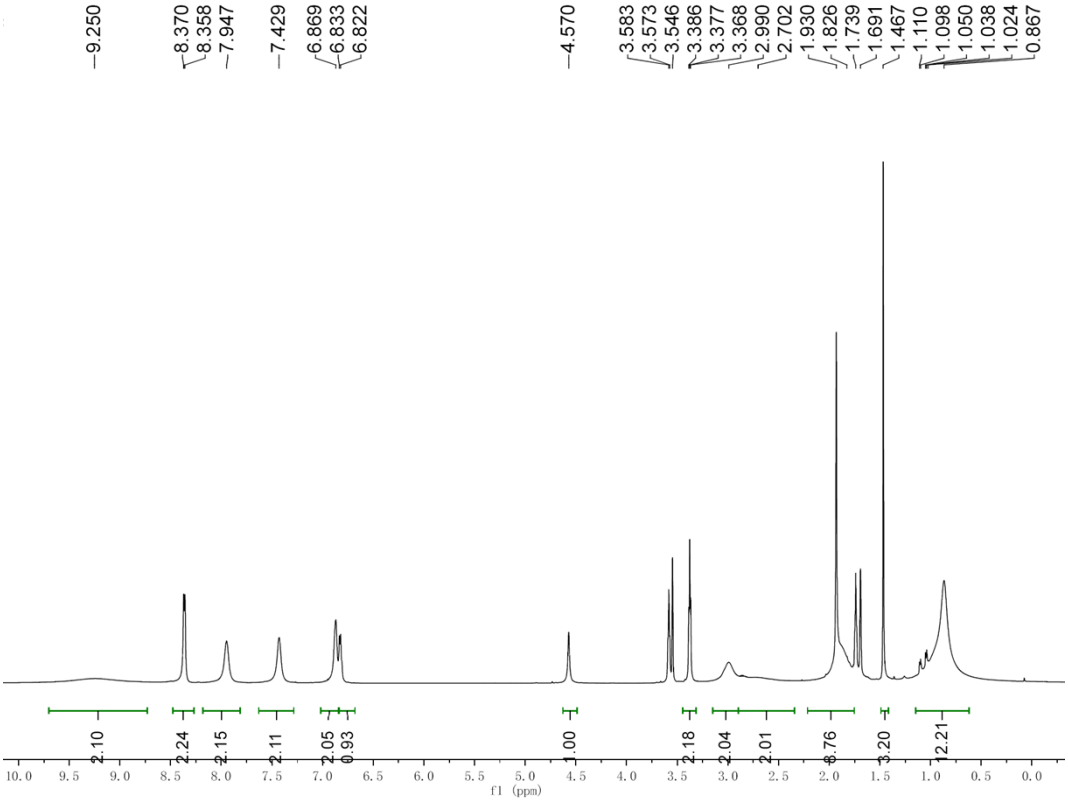

Fig. S6 ^{1}H NMR spectrum of compound **2**

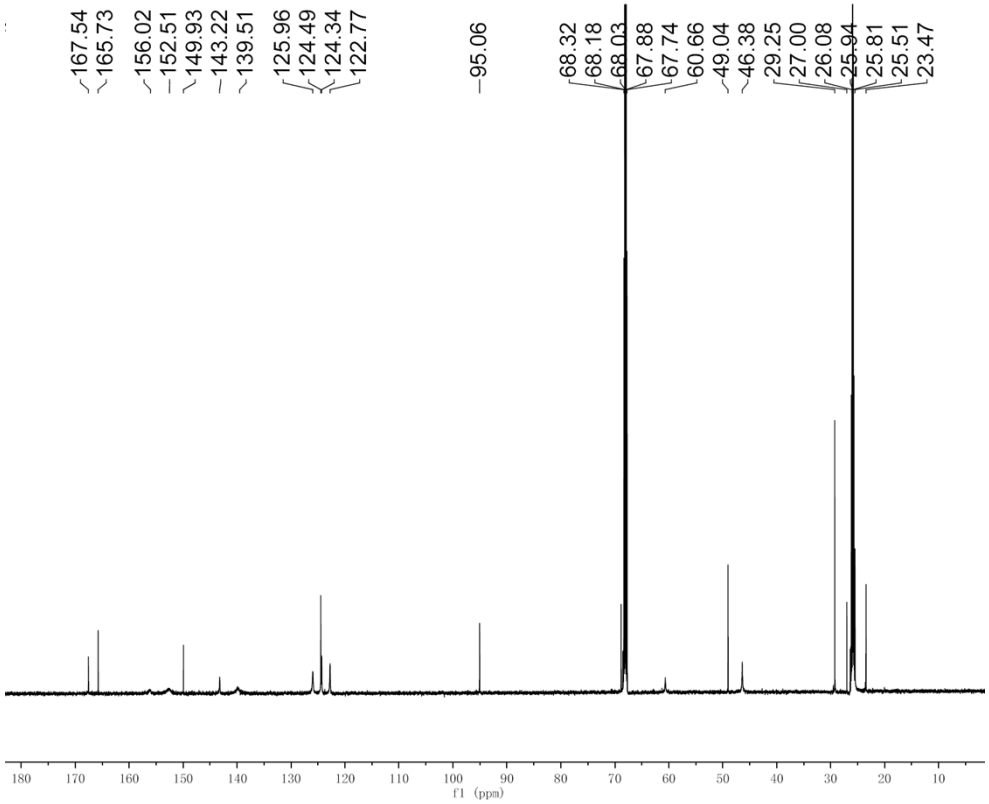

Fig. S7 ^{13}C NMR spectrum of compound **2**

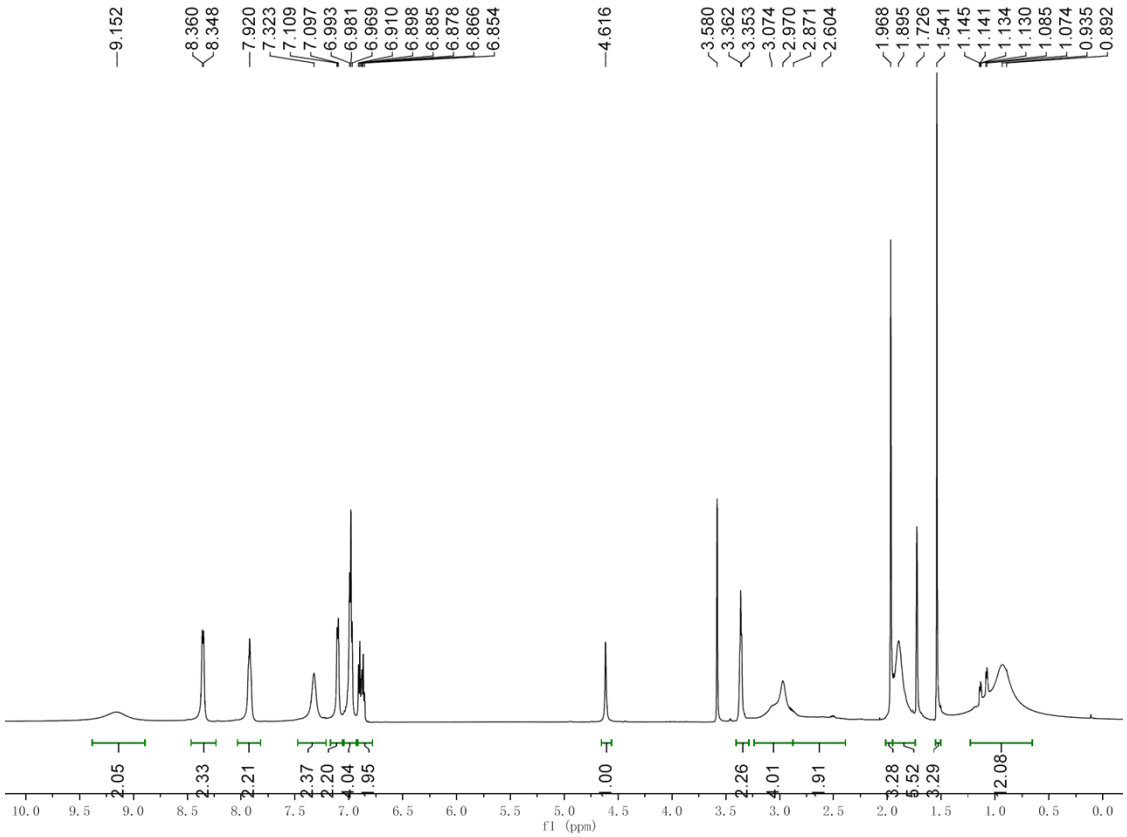

Fig. S8 ^{1}H NMR spectrum of compound **3**

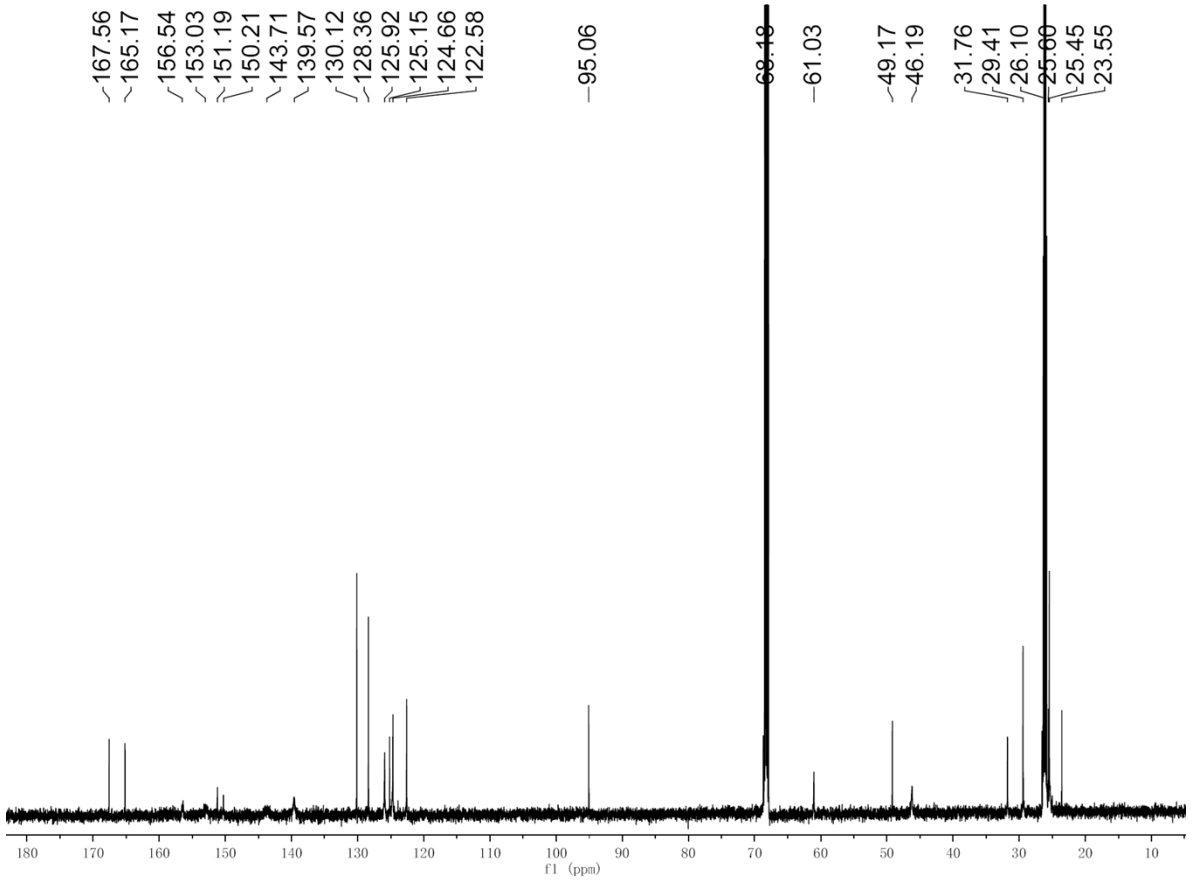

Fig. S9 ^{13}C NMR spectrum of compound **3**

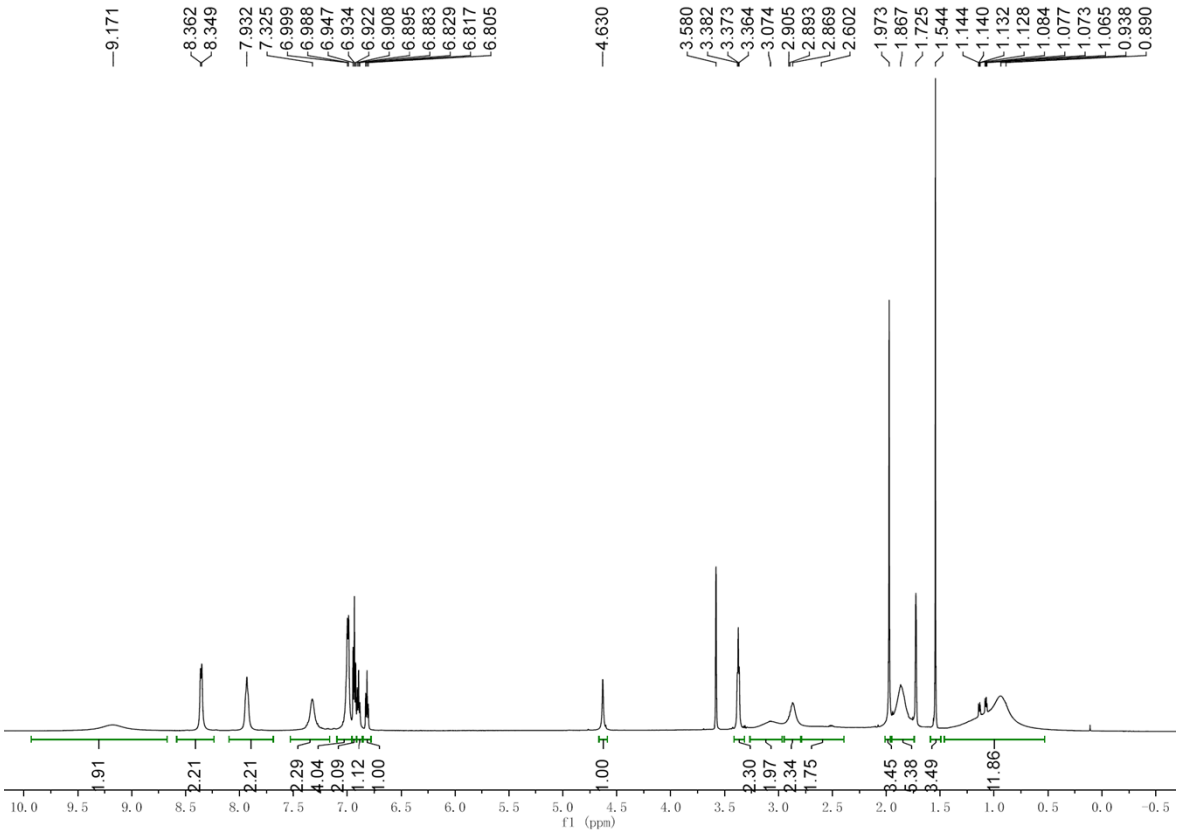

Fig. S10 ^{1}H NMR spectrum of compound **4**

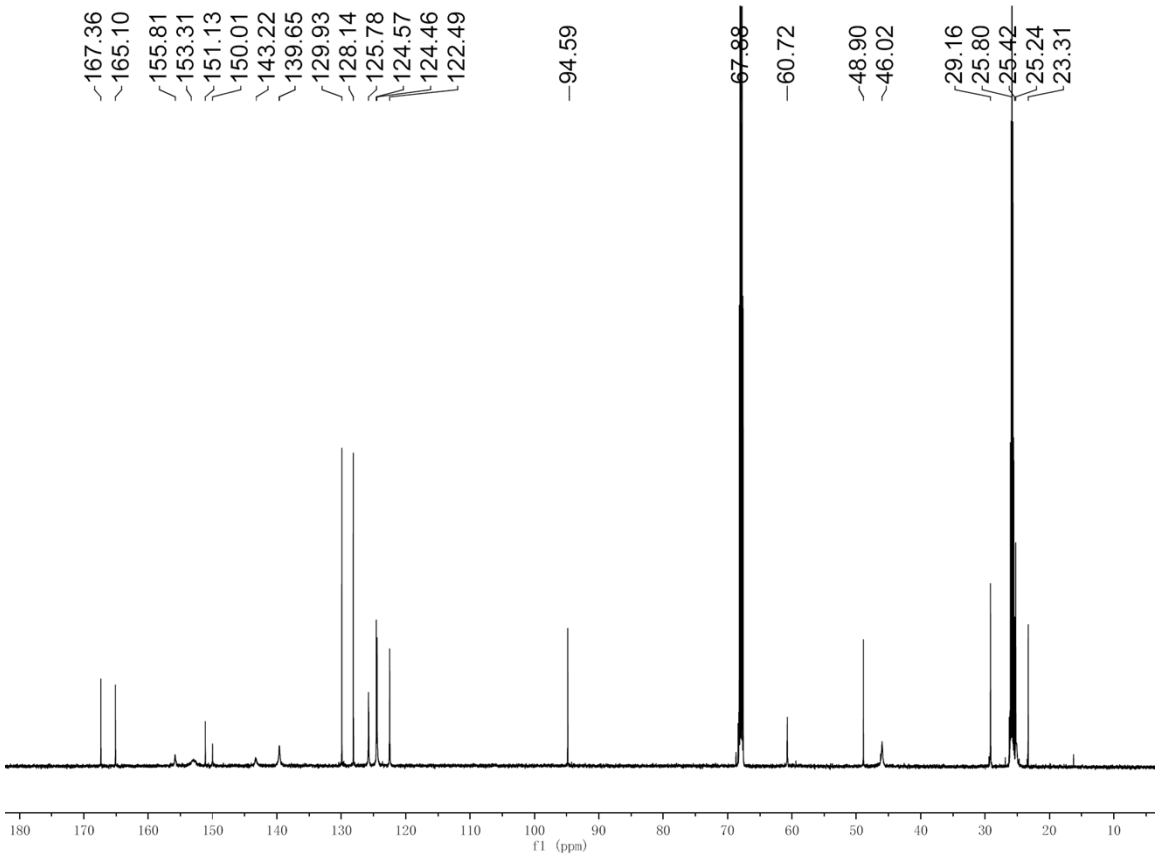

Fig. S11 ^{13}C NMR spectrum of compound **4**

IR SPECTRA

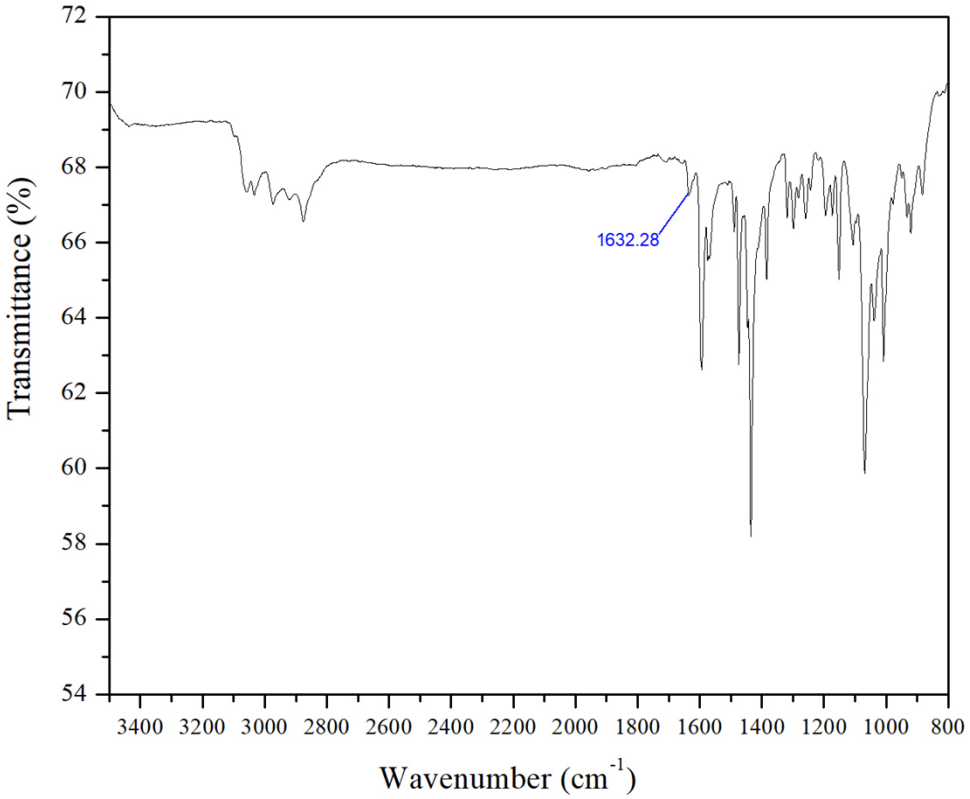

Fig. S12 IR spectrum of compound **7**

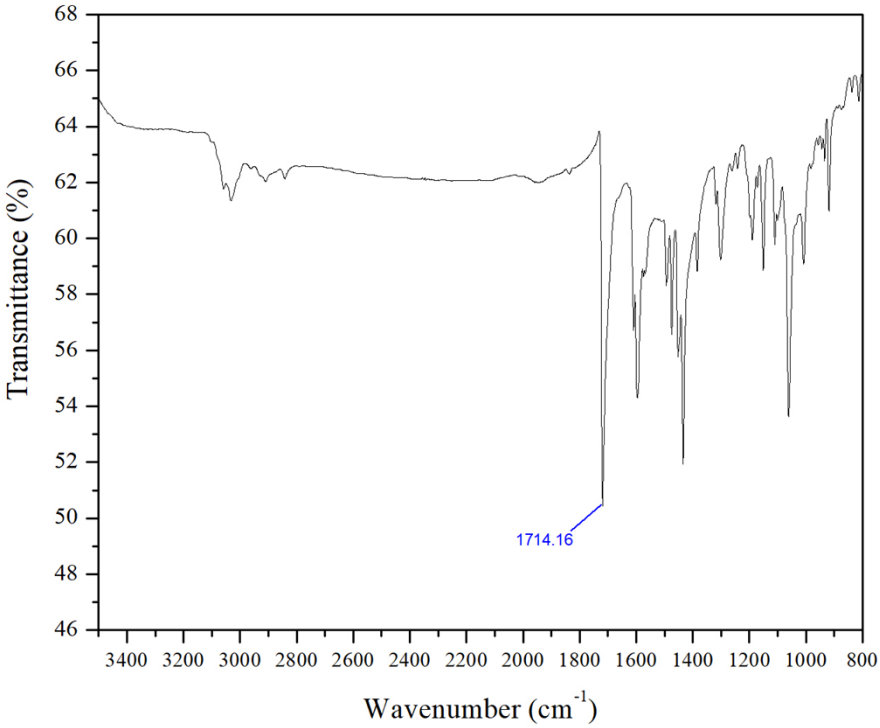

Fig. S13 IR spectrum of compound **8**