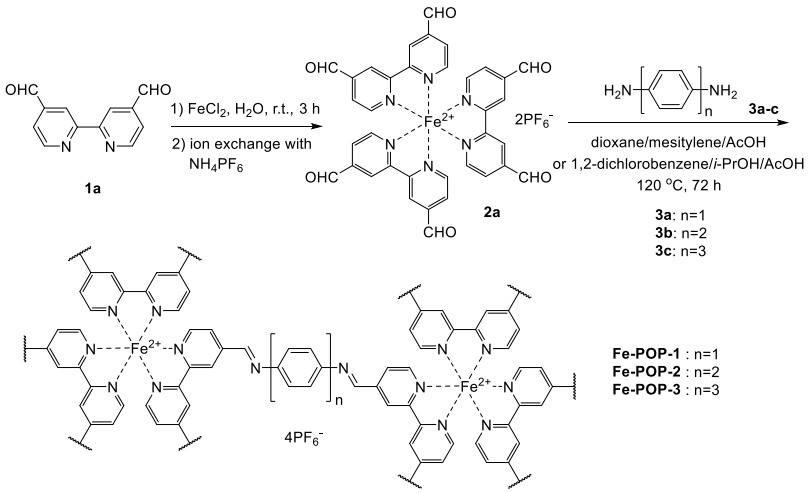
Electronic Supplementary Material (ESI) for Journal of Materials Chemistry A.

General methods. Commercial reactants were used without further purification. Solvents were dried according to standard methods. ^{1}H NMR spectra were recorded by Bruker AVANCE III HD 400 MHz instrument, and were internally referenced to residual solvent signals (^{1}H NMR: $CDCl_3$ and DMSO-d_6 referenced at 7.26 and 2.54 ppm, respectively; ^{13}C NMR: DMSO-d_6 referenced at 40.45 ppm). Gas chromatography was conducted on an Agilent 6890. Single crystal X-ray diffraction was recorded on a Bruker SMART CCD diffractometer. UV-visible spectra were recorded with a PerkinElmer Lambda 365 UV-Visible spectrometer. Fluorescence spectra were recorded with a PerkinElmer LS-55 fluorescence spectrometer. Fourier Transform Infrared spectra were recorded with a ThermoFisher Nicolet iS10 FT-IR spectrometer. Thermogravimetric analysis (TGA) profiles were recorded on a TGA 8000 thermogravimetric analyzer. Nitrogen adsorption experiments were recorded on a Micromeritics Tristar II 3020 analyzer. X-ray photoelectron spectra (XPS) were recorded on a PHI 5000C&PHI5300 spectrometer. Scanning electron microscopic (SEM) images were obtain on a Phenom Prox microscope. Energy dispersive spectra (EDS) were obtained on a Tecnai G2 F20 S-Twin microscope. ICP-AES analysis were obtained on a PE-8000 inductively coupled plasma emission spectrometer. The crystal data of complex **2a** has been deposited in Cambridge Crystallographic Data Centre (CCDC deposit no. 2040861).



Compound 2a. Compound **1a** (636.6 mg, 3.0 mmol) and ferrous chloride (126.7 mg, 1.0 mmol) were dissolved in deionized water (15 mL). The mixture was stirred for 3 h at room temperature. Saturated aqueous solution of NH_4PF_6 was added dropwise to the mixture till no more precipitate formed. The precipitate was filtered to afford the crude product, which was dissolved in CH_3CN to prepare a saturated solution. Ethyl ether was added dropwise to the solution, and the precipitate formed was filtrated and washed with cold diethyl ether to give **2a** as a bronze solid (834 mg, 85%). ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.25 (s, 6H), 9.06 (s, 6H), 8.86 (s, 6H), 7.96 (s, 6H). ^{13}C NMR (100 MHz, DMSO-d_6): δ 193.97, 156.51, 151.73, 143.49, 123.36, 120.04. FT-IR (KBr) 1707, 1617, 1559, 1478, 1416, 1390, 1380, 1314, 1232, 1198, 1181, 843 cm^{-1}. The structure of **2a** was further confirmed by its X-ray crystal structure (CCDC deposit no. 2040861). The single crystals were grown by evaporating the solution in acetonitrile.

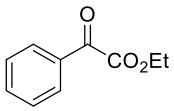
Fe-POP-1. Compounds **2a** (32.7 mg, 0.033 mmol) and **3a** (10.8 mg, 0.1 mmol) were dissolved in a mixture of 1,4-dioxane/mesitylene (19/1, v/v) (2 mL) and aqueous acetic acid (6 M, 0.2 mL) in a 10 mL Schlenk tube. The tube was sealed after being degassed in a liquid nitrogen

bath for 10 min and then kept at 120 ºC for 72 h to yield a dark purple solid at the bottom of the tube. After cooling to room temperature, the precipitate was filtered and washed with DMSO and further extracted with THF in a Soxhlet extractor. After being dried under vacuum at 40 ºC for 12 h, the POP was obtained as a dark purple powder (30.7 mg, 77%), which was insoluble in common solvents including water, DMSO, DMF, acetone or acetonitrile.

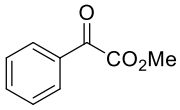
Fe-POP-2. Compounds **2a** (32.7 mg, 0.033 mmol) and **3b** (18.4 mg, 0.1 mmol) were dissolved in a mixture of 1,4-dioxane/mesitylene (3/1, v/v) (2 mL) and aqueous acetic acid (6 M, 0.2 mL) in a 10 mL Schlenk tube. The tube was sealed after being degassed in a liquid nitrogen bath for 10 min and then kept at 120 ºC for 72 h and then cooled to room temperature. The dark purple precipitate formed was filtered and washed with DMSO. The solid was then extracted in a Soxhlet extractor with THF and then dried under vacuum at 40 ºC for 12 h to give **Fe-POP-2** as a dark purple powder (40.3 mg, 85%). The product was insoluble in common solvents including water, DMSO, DMF, acetone or acetonitrile.

Fe-POP-3. Compounds **2a** (32.7 mg, 0.033 mmol) and **3c** (26.0 mg, 0.1 mmol) were dissolved in a mixture of 1,2-dichlorobenzene/isopropanol (1/3, v/v) (2 mL) and aqueous acetic acid (6 M, 0.2 mL) in a 10 mL Schlenk tube. The tube was sealed after being degassed in a liquid nitrogen bath for 10 min and then kept at 120 ºC for 72 h to yield a purple solid. After cooling to room temperature, the solid was filtered, washed with DMSO, extracted in a Soxhlet extractor with THF and dried under vacuum at 40 ºC for 12 h to give **Fe-POP-3** as a purple powder (45.2 mg, 82%), which was insoluble in common solvents including water, DMSO, DMF, acetone or acetonitrile.

General method for visible-light-driven oxidation reaction of benzyl halides. Benzyl halide **4** (1 mmol, 1.0 equiv.), **Fe-POP-3** (16.5 mg, 0.01 mmol, 0.01 equiv.), 4-methoxypyridine (21.8 mg, 0.2 mmol, 0.2 equiv.) and Li_2CO_3 (73.9 mg, 1.0 mmol, 1.0 equiv.) were dissolved in DMAc (5 mL). The heterogeneous catalyst was dispersed via ultrasound and a needle was inserted into the reaction flask to provide air. The mixture was stirred under the irradiation of two blue LED lights for 24 h at the distance of 8 cm. A powerful fan was used to keep the system cool. After reaction, 5 mL HCl (aq., 1 M) was added to the mixture. The mixture was extracted with ethyl acetate (10 mL × 3) and the organic phase was combined. The solution was then washed with deionized water (15 mL × 3) and brine (15 mL) and concentrated *in vacuo*. The product was purified by flash column chromatography on silica gel (hexane/ether acetate) to give **5**.

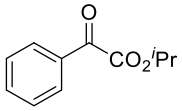


Compound 5a. Compound **4a** (243.1 mg) was used to give **5a** (125 mg, 70%), which was purified by flash column chromatography (hexane/ether acetate=30/1, v/v) as pale-yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02-7.99 (m, 2H), 7.65 (tt, J = 7.4, 1.2 Hz, 1H), 7.51 (t, J = 7.8 Hz, 2H), 4.45 (q, J = 7.2 Hz, 2H), 1.42 (t, J = 7.2 Hz, 3H).

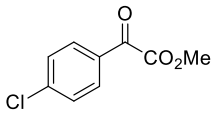


Compound 5b. Compound **4b** (229 mg) was used to give **5b** (144 mg, 87%), which was purified by flash column chromatography (hexane/ether acetate=40/1, v/v) as colorless oil. ^{1}H

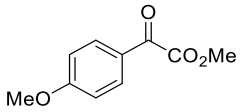
NMR (400 MHz, $CDCl_3$) δ 8.01 (dd, *J* = 8.4, 1.2 Hz, 2H), 7.66 (tt, *J* = 7.6, 1.3 Hz, 1H), 7.51 (t, *J* = 7.8 Hz, 2H), 3.98 (s, 3H).



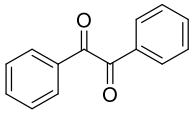
Compound 5c. Compound **4c** (259.2 mg) was used to give **5c** (198.4 mg, 93%), purified by flash column chromatography (hexane/ether acetate = 40/1, v/v) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.0 Hz, 2H), 7.65 (t, *J* = 7.2 Hz, 1H), 7.51 (t, *J* = 7.8 Hz, 2H), 5.37-5.27 (m, 1H), 1.41 (d, *J* = 6.0 Hz, 6H).



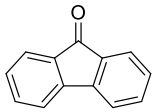
Compound 5d. Compound **4d** (264 mg) was used to give **5d** (160 mg, 81%), purified by flash column chromatography (hexane/ether acetate = 50/1, v/v) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (dt, *J* = 8.8, 2.2 Hz, 2H), 7.48 (dt, *J* = 8.8, 2.1 Hz, 2H), 3.97 (s, 3H).



Compound 5e. Compound **4d** (264 mg) was used to give **5c** (160 mg, 81%), purified by flash column chromatography (hexane/ether acetate = 40/1, v/v) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (dt, *J* = 9.2, 2.4 Hz, 2H), 6.97 (dt, *J* = 9.2, 2.4 Hz, 2H), 3.96 (s, 3H), 3.90 (s, 3H).

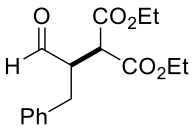


Compound 5f. Compound **4f** (275.2 mg) was used to give **5f** (202 mg, 96%), purified by flash column chromatography (hexane/ether acetate = 50/1, v/v) as yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99-7.97 (m, 2H), 7.66 (tt, *J* = 7.4, 1.2 Hz, 1H), 7.52 (t, *J* = 7.8 Hz, 2H).

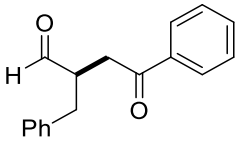


Compound 5g. Compound **4g** (245.1 mg) was used to give **5g** (160.4 mg, 88%), purifed by flash column chromatography (hexane/ether acetate = 50/1, v/v) as yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, *J* = 7.6 Hz, 1H), 7.54-7.47 (m, 2H), 7.29 (td, *J* = 7.4, 1.2 Hz, 1H).

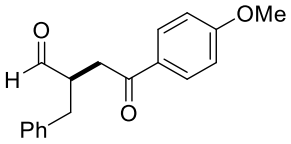
General method for visible-light-driven enantioselective α-alkylation of aldehydes. Aldehyde **6** (1.0 mmol, 2.0 equiv.), bromide **7** (0.5 mmol, 1.0 equiv.), chiral organocatalyst **8** (32 mg, 0.1 mmol, 0.2 equiv.), **Fe-POP-3** (8.7 mg, 0.005 mmol, 0.01 equiv.) and 2,6-lutine (107 mg, 1.0 mmol, 2.0 equiv.) were dissolved in anhydrous DMF (1 mL). The reaction flask was sealed after the heterogeneous catalyst was dispersed via ultrasound and the mixture was protected by N_2. The mixture was stirred with irradiation of two blue LED lights for 24 h. A fan was used to keep the system cool. After reaction, 5 mL HCl (aq., 1 M) was added to the mixture. The mixture was extracted with ethyl acetate (10 mL × 3) and the organic phase was combined. The solution was then washed with deionized water (15 mL × 3) and brine (15 mL) and concentrated *in vacuo*. The product was purified by flash column chromatography on silica gel (hexane/ether acetate) to give **9**.



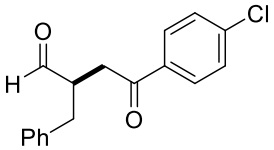
Compound 9a. Compounds **6a** (134.2 mg) and **7a** (119.5 mg) were used to give **9a** (121 mg, 83% yield, 95% ee), purified flash column chromatography (hexane/ether acetate = 11/1, v/v) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.75 (s, 1H), 7.30-7.15 (m, 5H), 4.22-4.14 (m, 4H), 3.65 (d, *J* = 8.0 Hz, 1H), 3.36 (q, *J* = 7.3 Hz, 1H), 3.10 (dd, *J* = 14.0, 7.6 Hz, 1H), 2.80 (dd, *J* = 14.0, 7.6 Hz, 1H), 1.24 (t, *J* = 7.2 Hz, 6H). The enantiomeric excess was determined by chiral HPLC with IC chiral column (hexane/isopropanol=90/10; flow 1 mL·min^{-1}); (*S*)-isomer: t*r* = 14.38 min, (*R*)-isomer: t*r* = 17.08 min.



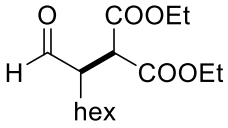
Compound 9b. Compounds **6a** (134.2 mg) and **7b** (99.5 mg) were used to give **9b** (108 mg, 85% yield, 92% ee), purified by flash column chromatography (hexane/ether acetate = 9/1, v/v) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.90 (s, 1H), 7.93-7.90 (m, 2H), 7.56 (tt, *J* = 7.4, 1.4 Hz, 1H), 7.46-7.42 (m, 2H), 7.33-7.29 (m, 2H), 7.25-7.30 (m, 3H), 3.47-3.37 (m, 2H), 3.17 (dd, *J* = 13.6, 6.2 Hz, 1H), 3.06-2.99 (m, 1H), 2.86-2.80 (m, 1H). The enantiomeric excess was determined by chiral HPLC with IC chiral column (hexane/isopropanol = 95/5; flow 1 mL·min^{-1}); (*S*)-isomer: t*r* = 14.68 min, (*R*)-isomer: t*r* = 16.06 min.



Compound 9c. Compounds **6a** (134.2 mg) and **7c** (116.7 mg) were used to give **9c** (113 mg, 80% yield, 89% ee), purified by flash column chromatography (hexane/ether acetate = 9/1, v/v) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.90 (s, 1H), 7.89 (d, *J* = 8.8 Hz, 2H), 7.32-7.19 (m, 5H), 6.91 (d, *J* = 8.8 Hz, 2H), 3.85 (s, 3H), 3.40-3.33 (m, 2H), 3.16 (dd, *J* = 14.0, 6.0 Hz, 1H), 2.99 (m, 1H), 2.82 (dd, *J* = 14.4, 7.6 Hz, 1H). The enantiomeric excess was determined by chiral HPLC with IC chiral column (hexane/isopropanol = 90/10; flow 1 mL·min^{-1}); (*S*)-isomer: t*r* = 33.54 min, (*R*)-isomer: t*r* = 37.53 min.

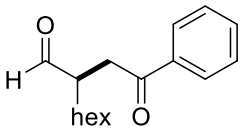


Compound 9d. Compounds **6a** (134.2 mg) and **7d** (114.5 mg) were used to give **9d** (110.4 mg, 77% yield, 93% ee), purified by flash column chromatography (hexane/ether acetate = 10/1, v/v) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.88 (s, 1H), 7.83 (d, *J* = 8.4 Hz, 2H), 7.40 (d, *J* = 8.8 Hz, 2H), 7.32-7.18 (m, 5H), 3.42-3.34 (m, 2H), 3.17 (dd, *J* = 14.0, 6.0 Hz, 1H), 2.98-2.91 (m, 1H), 2.84-2.79 (m, 1H). The enantiomeric excess was determined by chiral HPLC with IC chiral column (hexane/isopropanol = 95/5; flow 1 mL·min^{-1}); (*S*)-isomer: t*r* = 13.80 min, (*R*)-isomer: t*r* = 15.40 min.

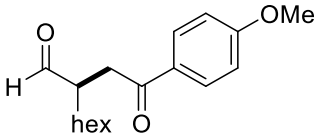


Compound 9e. Compounds **6b** (128.2 mg) and **7a** (99.5 mg) were used to give **9e** (123 mg, 86% yield, 93% ee), purified by flash column chromatography (hexane/ether acetate = 12/1, v/v) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.77 (s, 1H), 4.25-4.17 (m, 4H), 3.73 (d, *J* = 8.0 Hz, 1H), 3.10 (m, 1H), 1.75-1.66 (m, 1H), 1.61-1.54 (m, 1H), 1.39-1.24 (m, 16H), 0.89-0.85 (m, 3H).

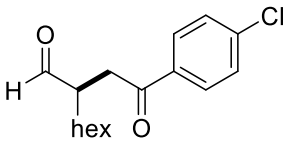
The enantiomeric excess was determined using the reported method (Ref. 58 in the main text): Compound **9e** (40 mg) was added to a mixture of 17.5 mg of (2*S*,4*S*)-(+)-pentanediol and 3 mg of p-toluenesulfonic acid monohydrate in DCM (2 mL). After 1 h, the consumption of the aldehyde was complete (monitored by TLC). The mixture was concentrated *in vacuo* and the enantiomeric excess of the title compound was determined by integrating the two ^{1}H NMR signals (both doublets) in $CDCl_3$ at 3.70 ppm (minor) and 3.67 ppm (major) arising from the resultant diastereomeric acetals.



Compound 9f. Compounds **6b** (128.2 mg) and **7b** (99.5 mg) were used to give **9f** (106 mg, 86% yield, 95% ee), purified by flash column chromatography (hexane/ether acetate = 8/1, v/v) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.81 (s, 1H), 7.97 (d, *J* = 8.0 Hz, 2H), 7.56 (t, *J* = 6.8 Hz, 1H), 7.46 (t, *J* = 7.2 Hz, 2H), 3.47 (dd, *J* = 18.4, 7.2 Hz, 1H), 3.12-2.99 (m, 2H), 1.83-1.74 (m, 1H), 1.57-1.49 (m, 1H), 1.37-1.27 (m, 8H), 0.87 (t, *J* = 6.0 Hz, 3H). The enantiomeric excess was determined by chiral HPLC with IC chiral column (hexane/isopropanol=95/5; flow 1 mL·min^{-1}); (*S*)-isomer: t*r* = 14.26 min, (*R*)-isomer: t*r* = 17.44 min.



Compound 9g. Compounds **6b** (128.2 mg) and **7c** (116.7 mg) were used to give **9g** (132 mg, 84% yield, 93% ee), purified by flash column chromatography (hexane/ether acetate = 9/1, v/v) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.82 (s, 1H), 7.95 (d, *J* = 9.2 Hz, 2H), 6.94 (d, *J* = 8.8 Hz, 2H), 3.87 (s, 3H), 3.41(dd, *J* = 17.2, 8.0 Hz, 1H), 3.11-3.04 (m, 1H), 2.99 (dd, *J* = 17.2, 4.8 Hz, 1H), 1.83-1.74 (m, 1H), 1.57-1.48 (m, 1H), 1.38-1.28 (m, 8H), 0.88 (t, *J* = 6.8 Hz, 3H). The enantiomeric excess was determined by chiral HPLC with IC chiral column (hexane/isopropanol=90/10; flow 1 mL·min^{-1}); (*S*)-isomer: t*r* = 26.48 min, (*R*)-isomer: t*r* = 30.94 min.



Compound 9h. Compounds **6b** (128.2 mg) and **7d** (114.5 mg) were used to give **9h** (109.5 mg,

79% yield, 93% ee), purified by flash column chromatography (hexane/ether acetate = 9/1, v/v) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.81 (s, 1H), 7.91 (d, *J* = 8.4 Hz, 2H), 7.44 (d, *J* = 8.8 Hz, 2H), 3.44 (dd, *J* = 18.0, 8.0 Hz, 1H), 3.14-3.07 (m, 1H), 2.95 (dd, *J* = 17.6, 4.8 Hz, 1H), 1.84-1.75 (m, 1H), 1.58-1.49 (m, 1H), 1.38-1.28 (m, 8H), 0.88 (t, *J* = 6.4 Hz, 3H). The enantiomeric excess was determined by chiral HPLC with IC chiral column (hexane/isopropanol = 95/5; flow 1 $mL \cdot min^{-1}$); (*S*)-isomer: t*r* = 12.32 min, (*R*)-isomer: t*r* = 14.44 min.

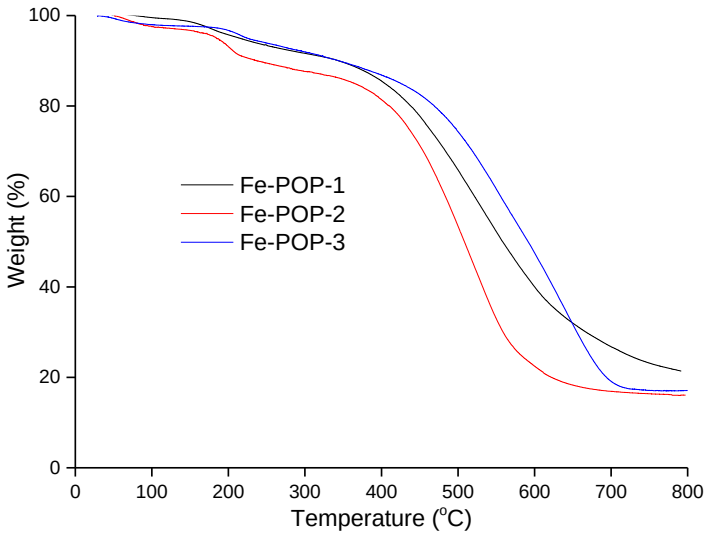


Fig. S1 Thermogravimetric profile of polymers **Fe-POP-1~3**.

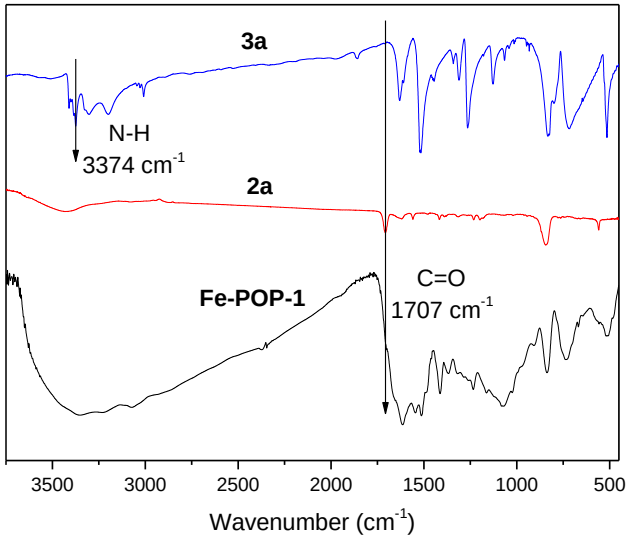


Fig. S2 FT-IR spectra of compound **2a**, **3a** and polymer **Fe-POP-1.**

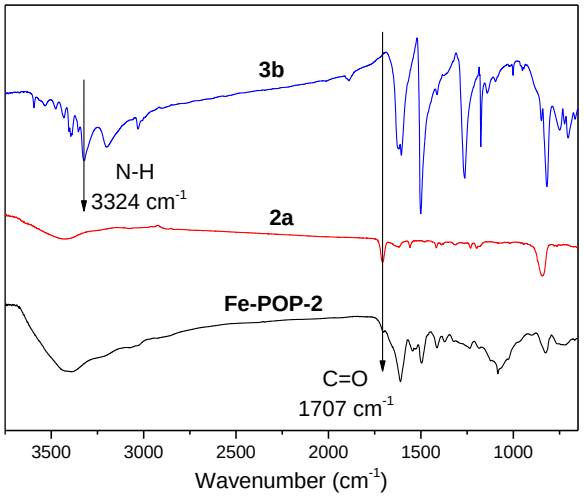


Fig. S3 FT-IR spectra of compound **2a**, **3b** and polymer **Fe-POP-2.**

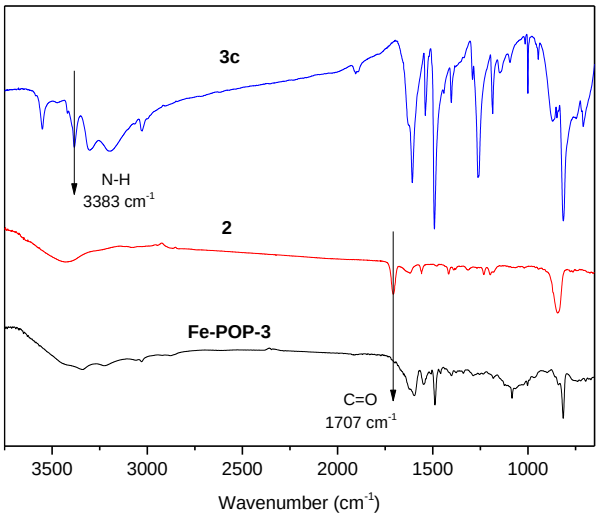


Fig. S4 FT-IR spectra of compound **2a**, **3c** and polymer **Fe-POP-3.**

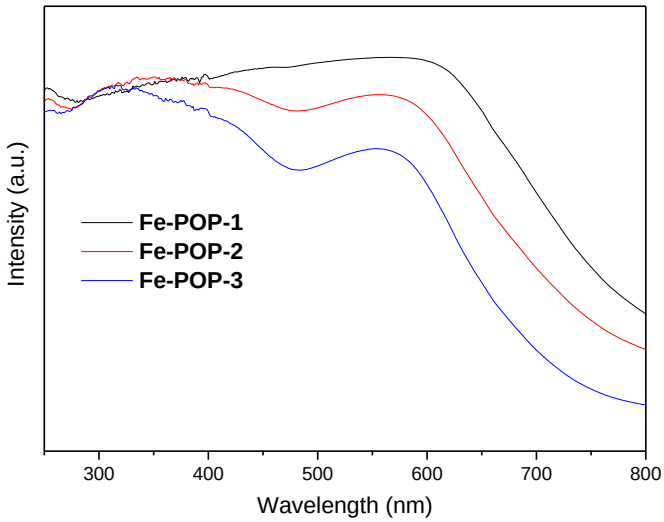


Fig. S5 Solid state UV-vis absorption spectra of **Fe-POP-1~3.**

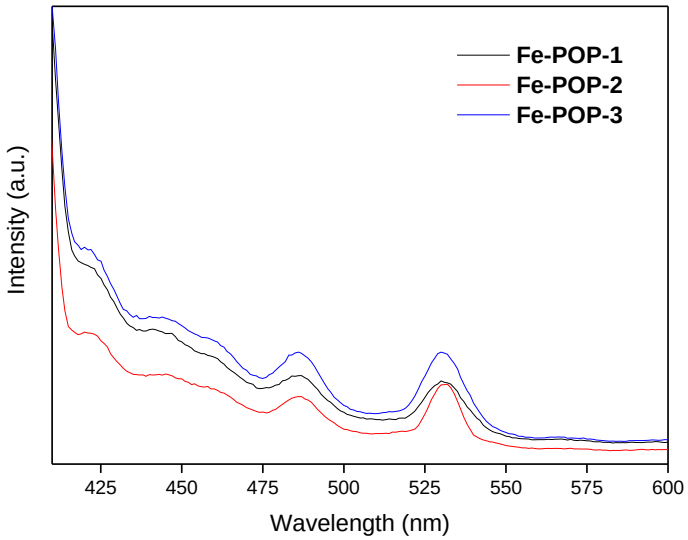


Fig. S6 Solid state fluorescence emission spectra of **Fe-POP-1~3.**

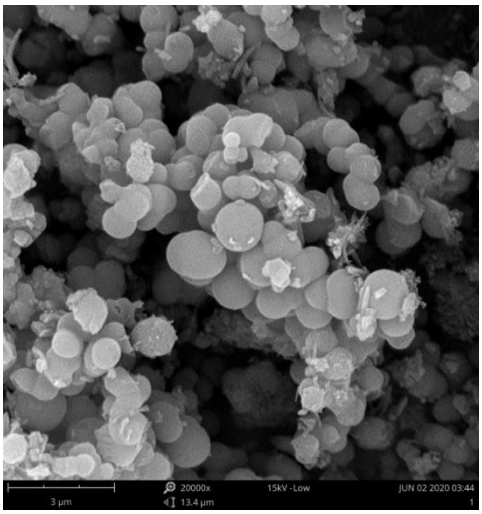


Fig. S7 Scanning electron microscope (SEM) image of **Fe-POP-1**.

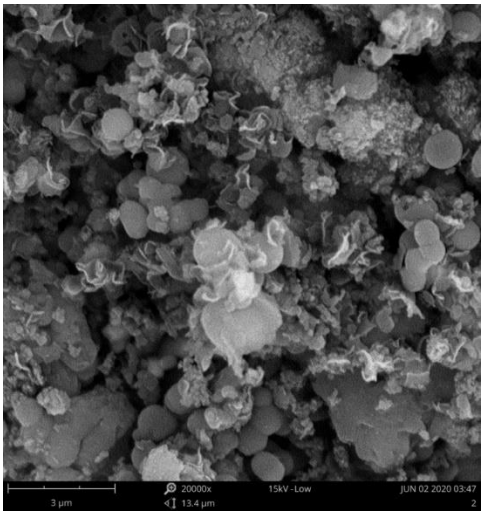


Fig. S8 Scanning electron microscope (SEM) image of **Fe-POP-2**.

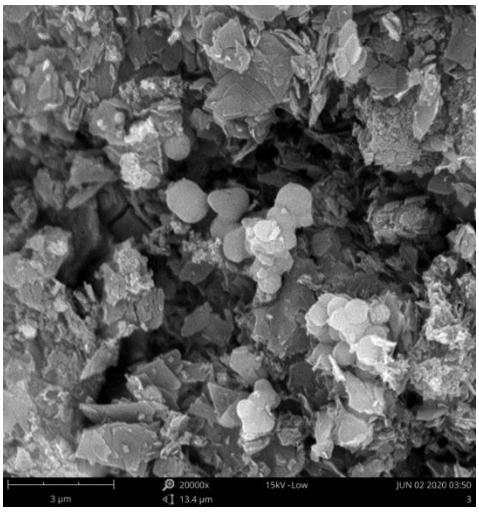


Fig. S9 Scanning electron microscope (SEM) image of **Fe-POP-3**.

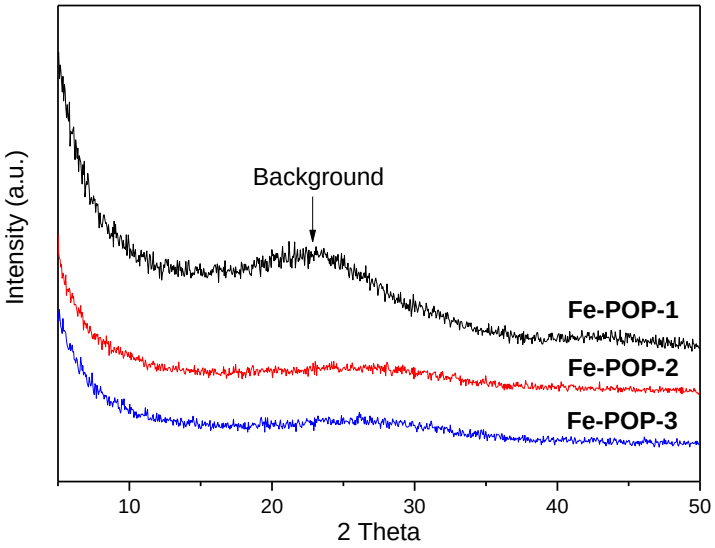


Fig. S10 PXRD spectra of **Fe-POP-1~3.**

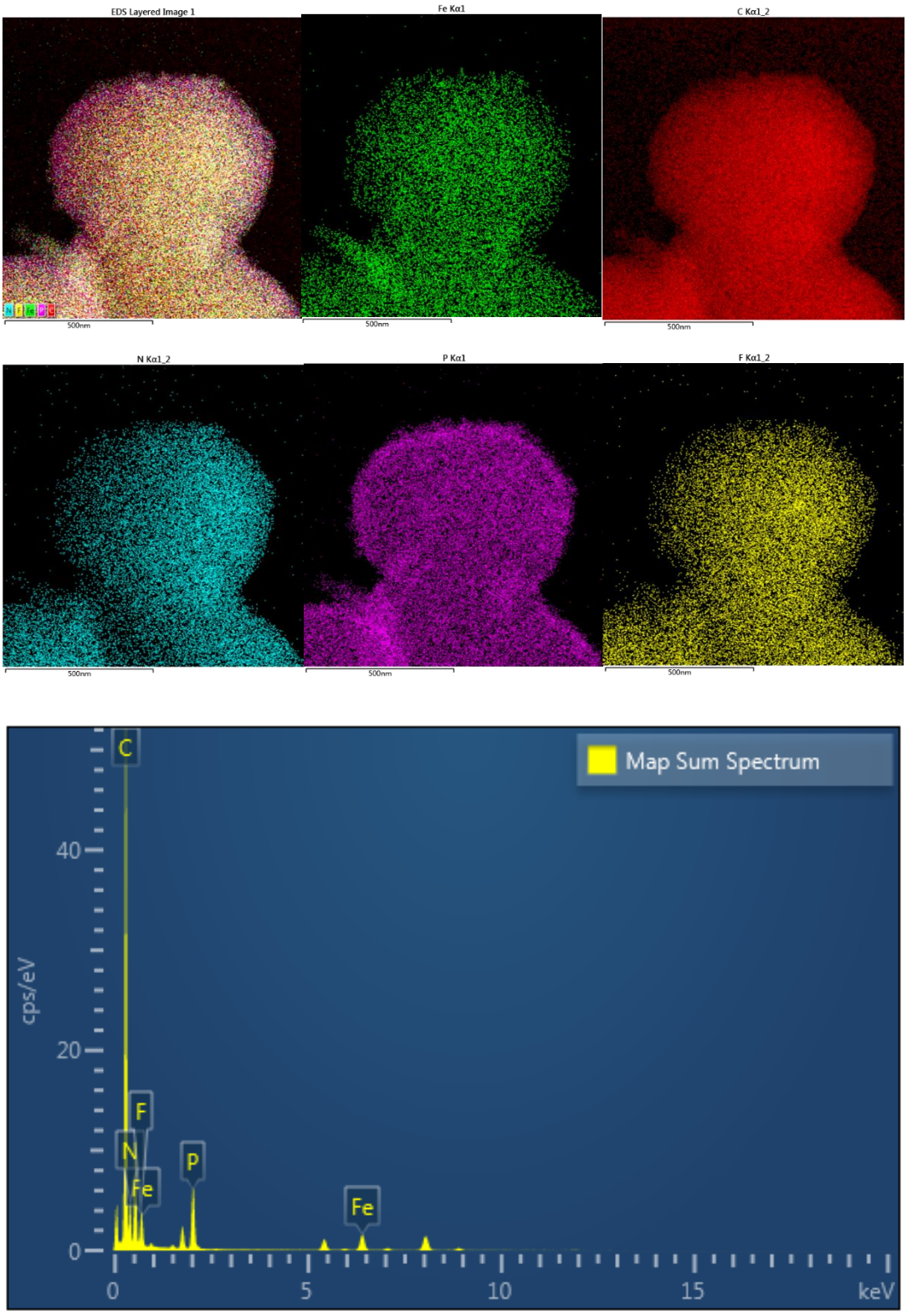


Fig. S11 Energy dispersive spectroscopic (EDS) images of polymer **Fe-POP-1**.

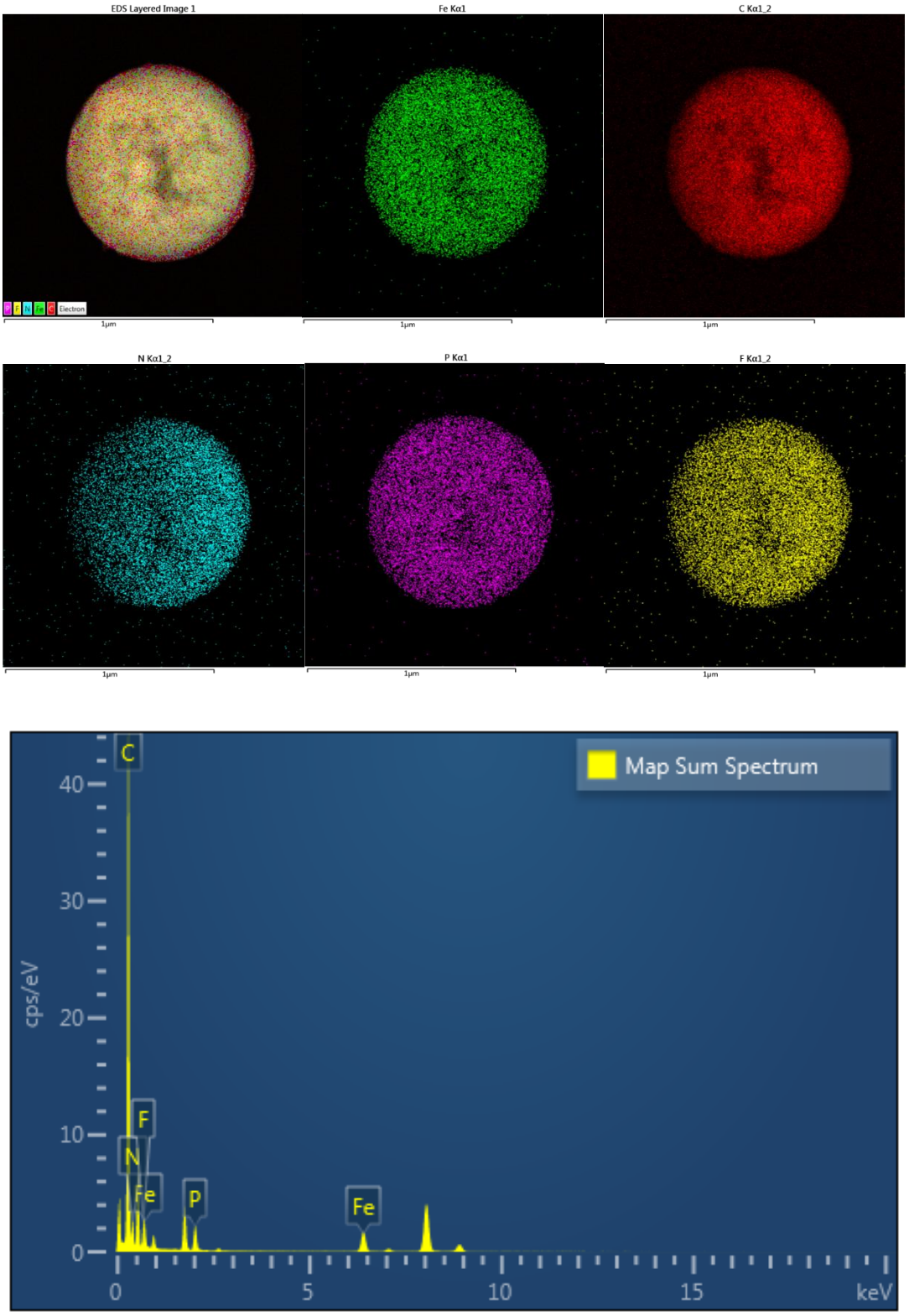


Fig. S12 Energy dispersive spectroscopic (EDS) images of polymer **Fe-POP-2**.

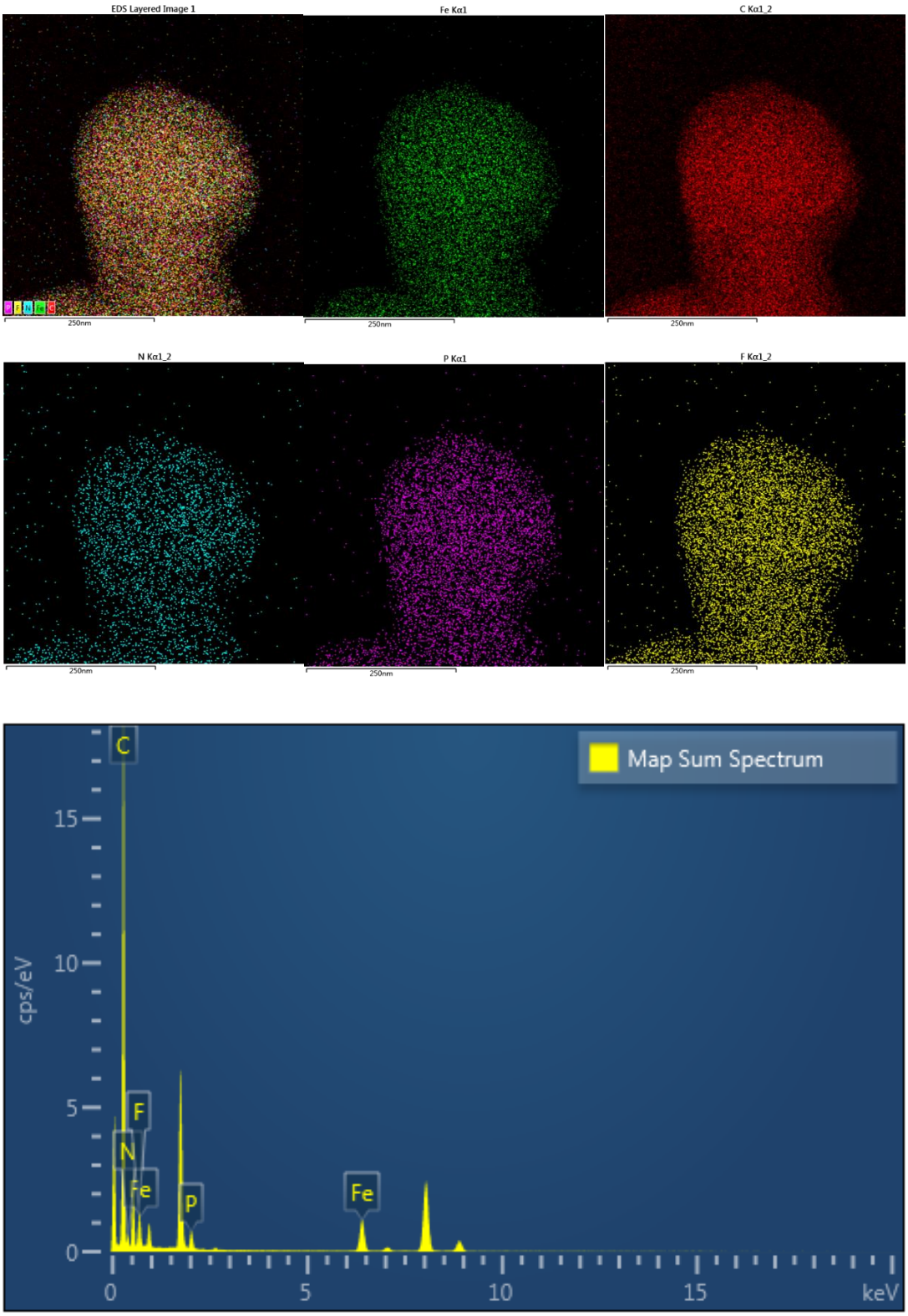


Fig. S13 Energy dispersive spectroscopic (EDS) images of polymer **Fe-POP-3**.

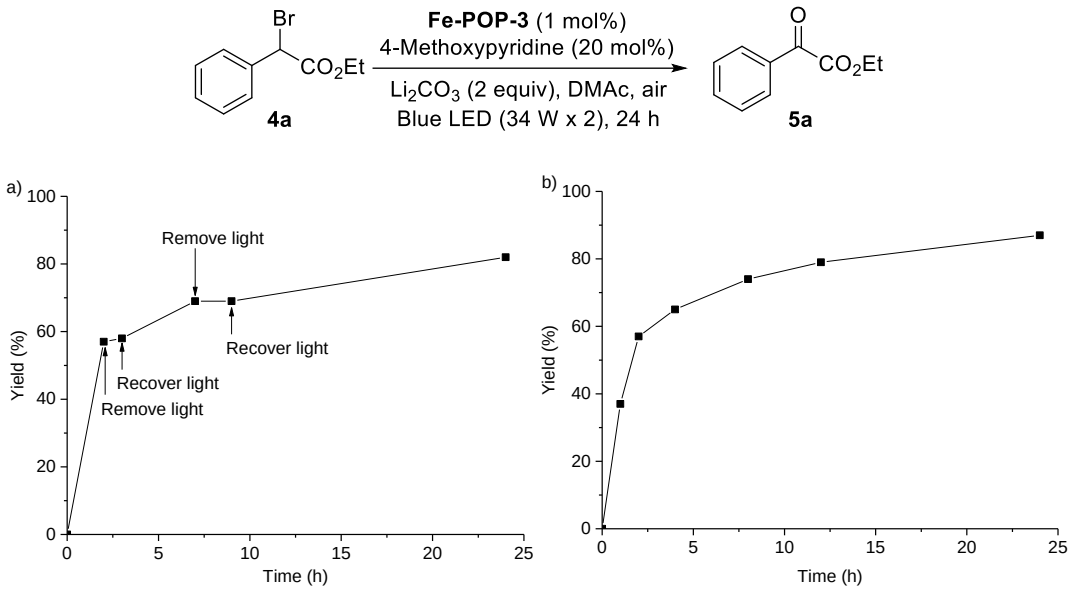


Fig. S14 a) On-off experiment and b) reaction progress curve for the oxidation of **4a** to produce **5a** in DMAc in the presence of **Fe-POP-3**.

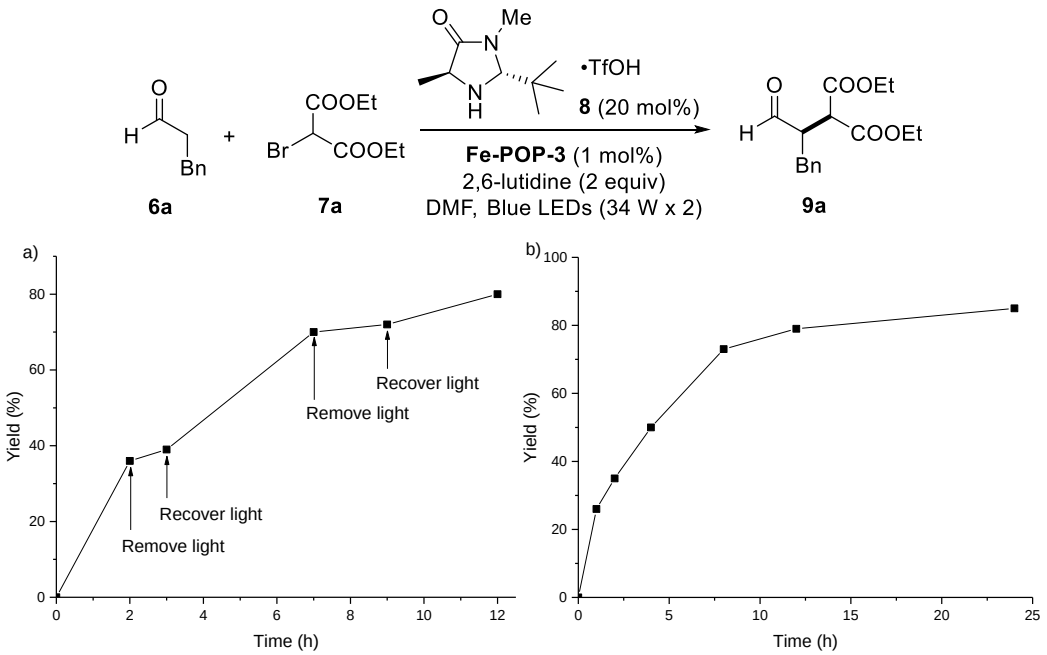


Fig. S15 a) On-off experiment and b) reaction progress curve for the alkylation of **6a** to produce **9a** in DMF in the presence of **Fe-POP-3**.

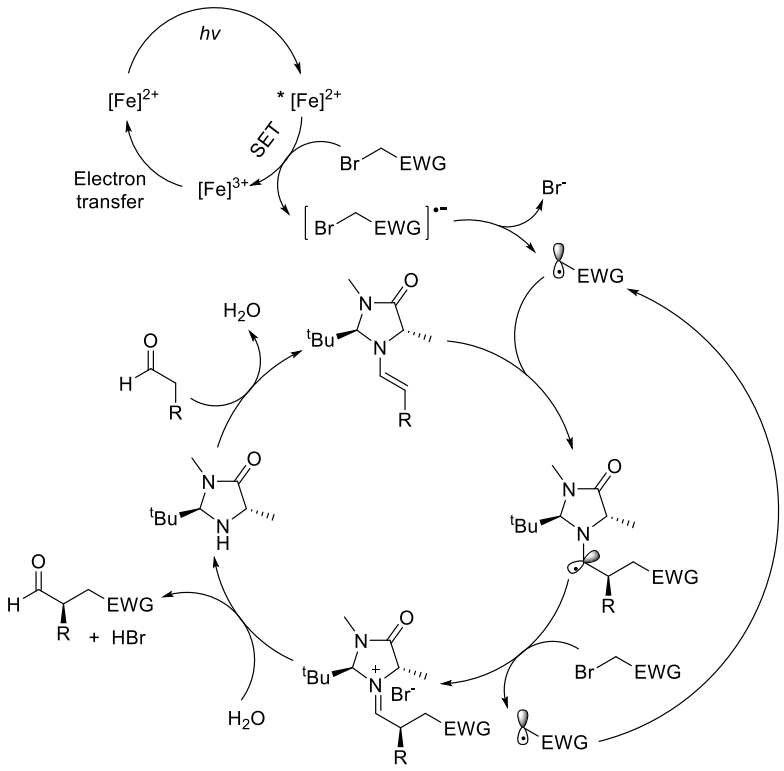


Fig. S16 Proposed mechanism for the photocatalytic enantioselective α-alkylation of aldehydes **6a-b** with bromides **7a-d** for the formation of compounds **9a-h** with polymer **Fe-POP-3** as heterogeneous catalyst.

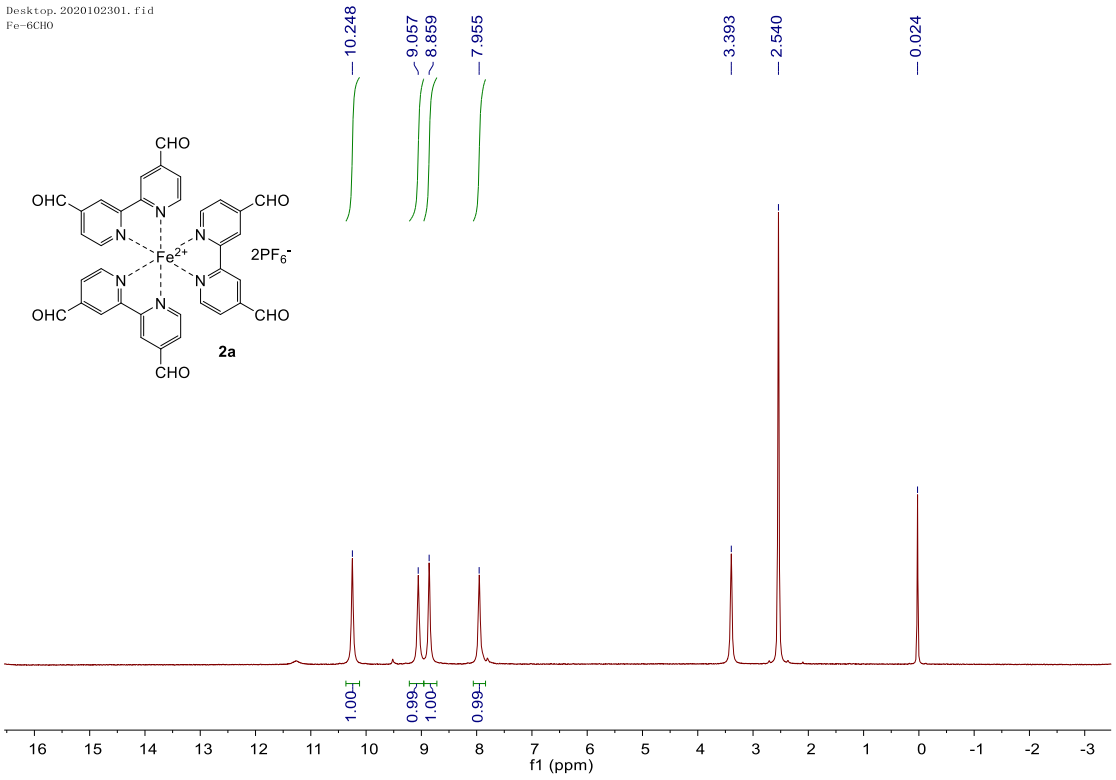


Fig. S17 ^{1}H NMR of compound **2a** in DMSO-d_6.

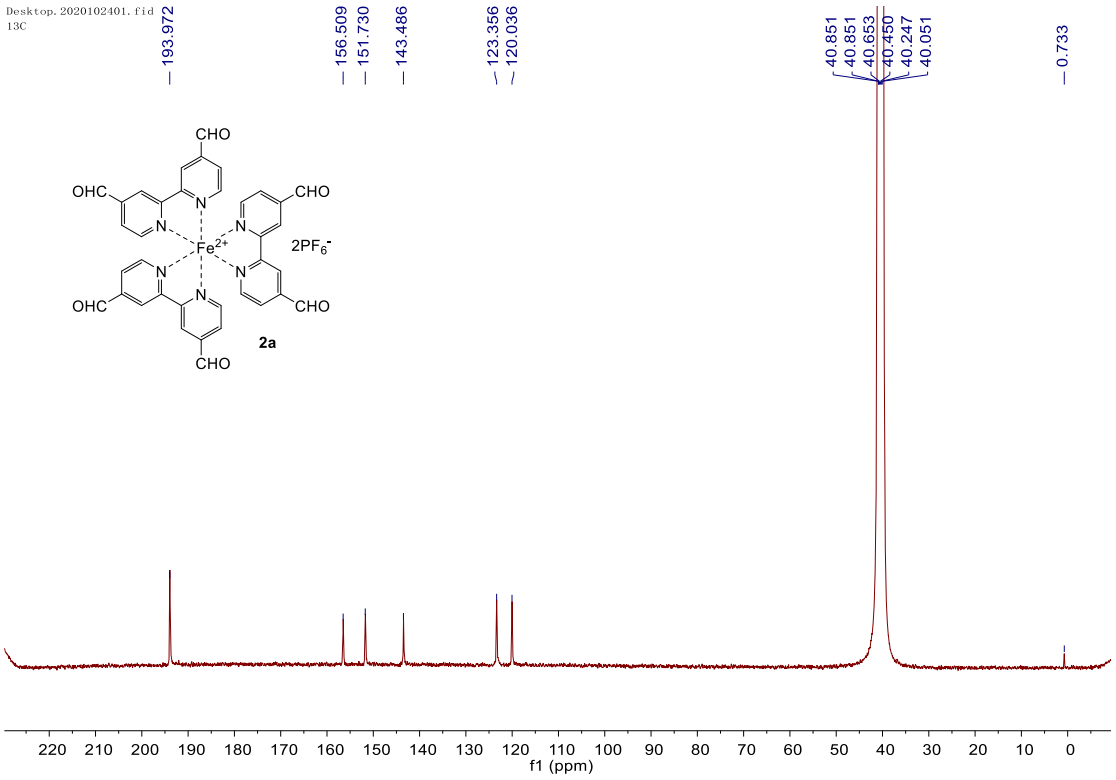


Fig. S18 ^{13}C NMR of compound **2a** in DMSO-d_6.

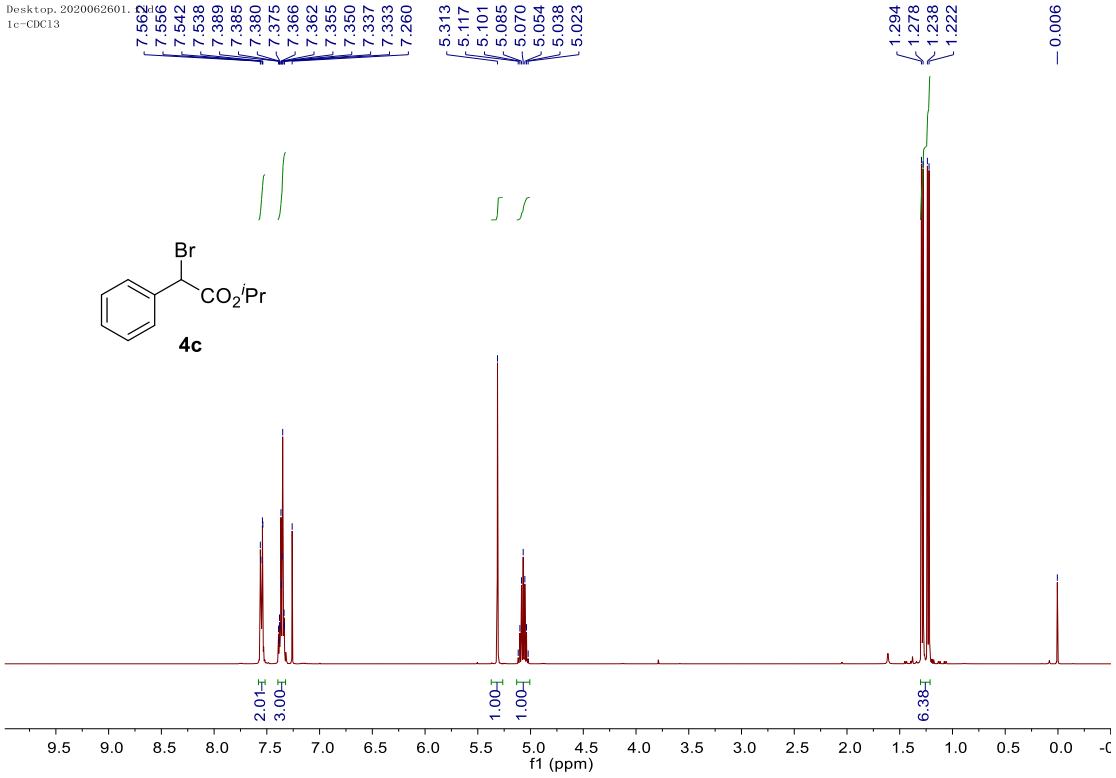


Fig. S19 ^{1}H NMR of compound **4c** in $CDCl_3$.

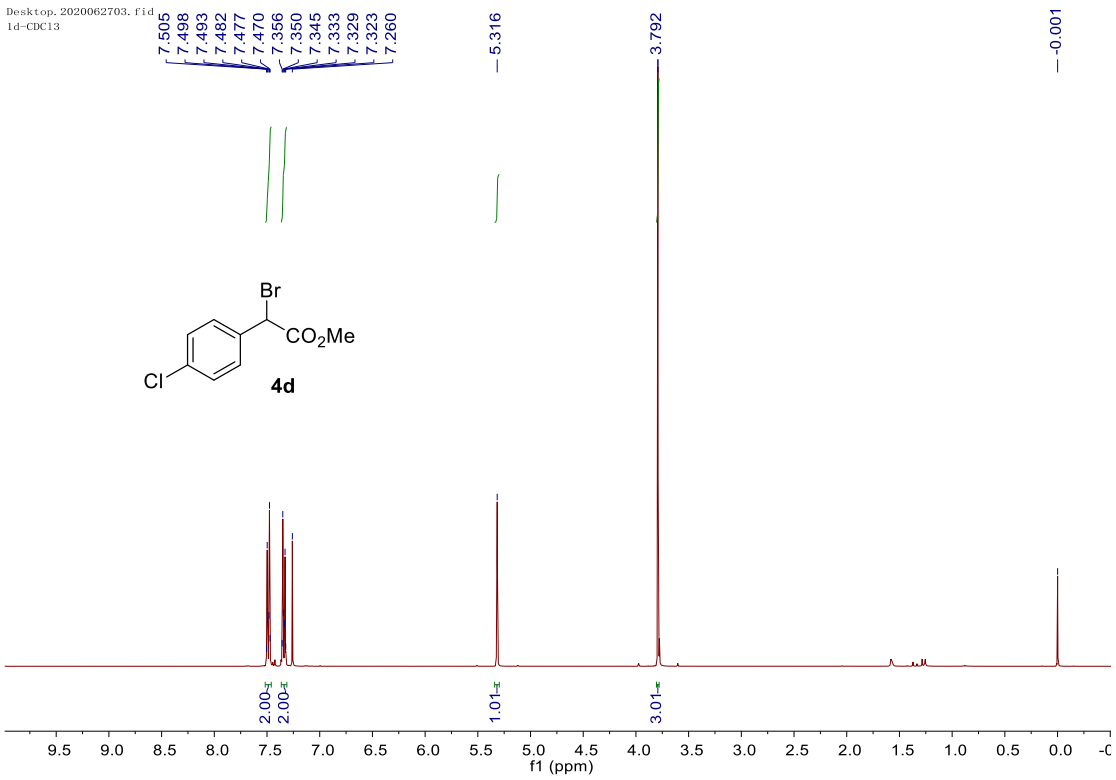


Fig. S20 ^{1}H NMR of compound **4d** in $CDCl_3$.

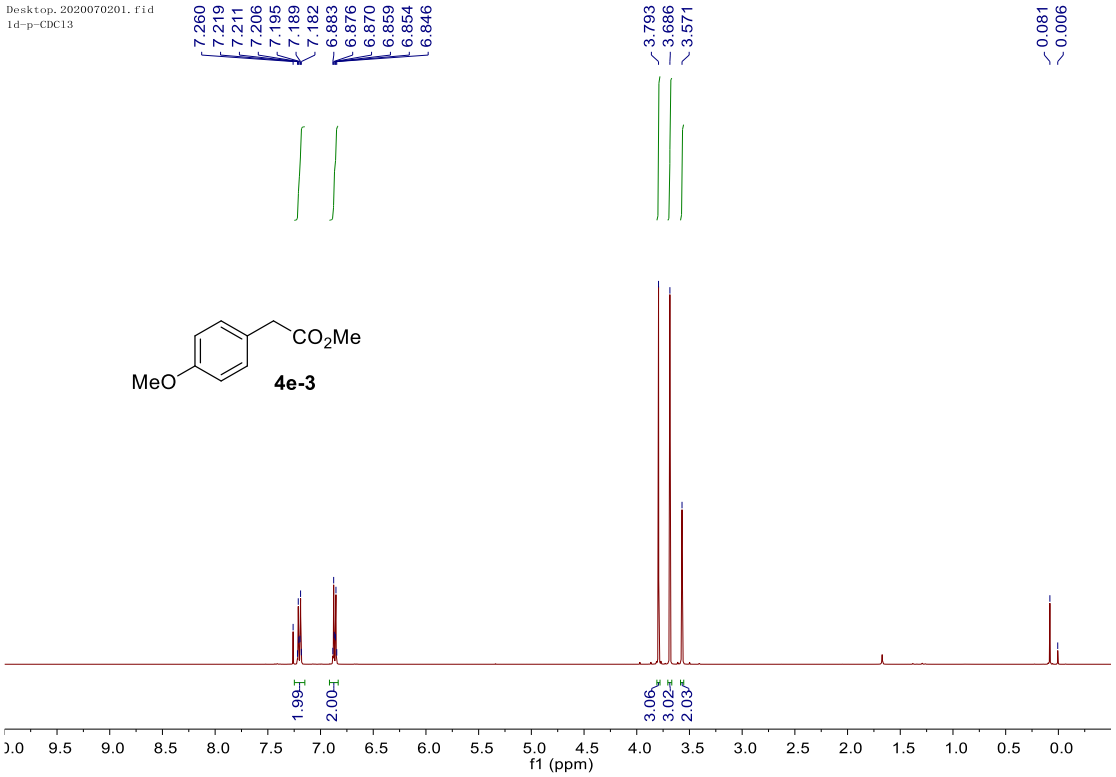


Fig. S21 ^{1}H NMR of compound **4e-3** in $CDCl_3$.

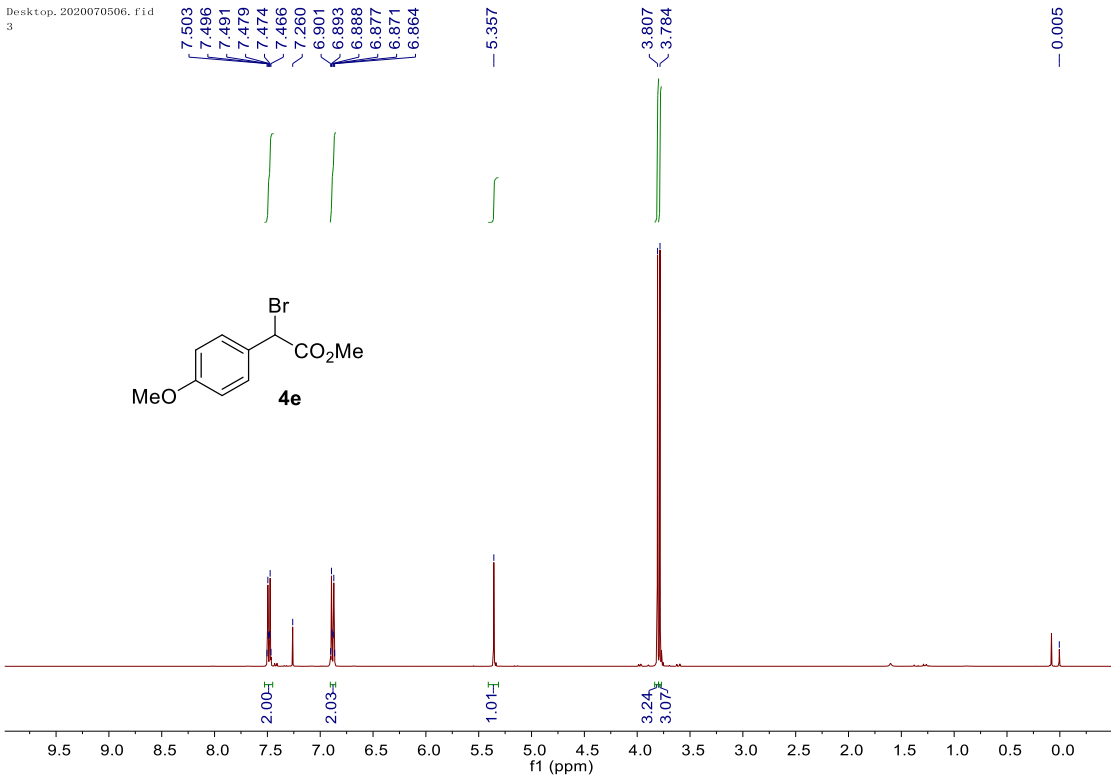


Fig. S22 ^{1}H NMR of compound **4e** in $CDCl_3$.

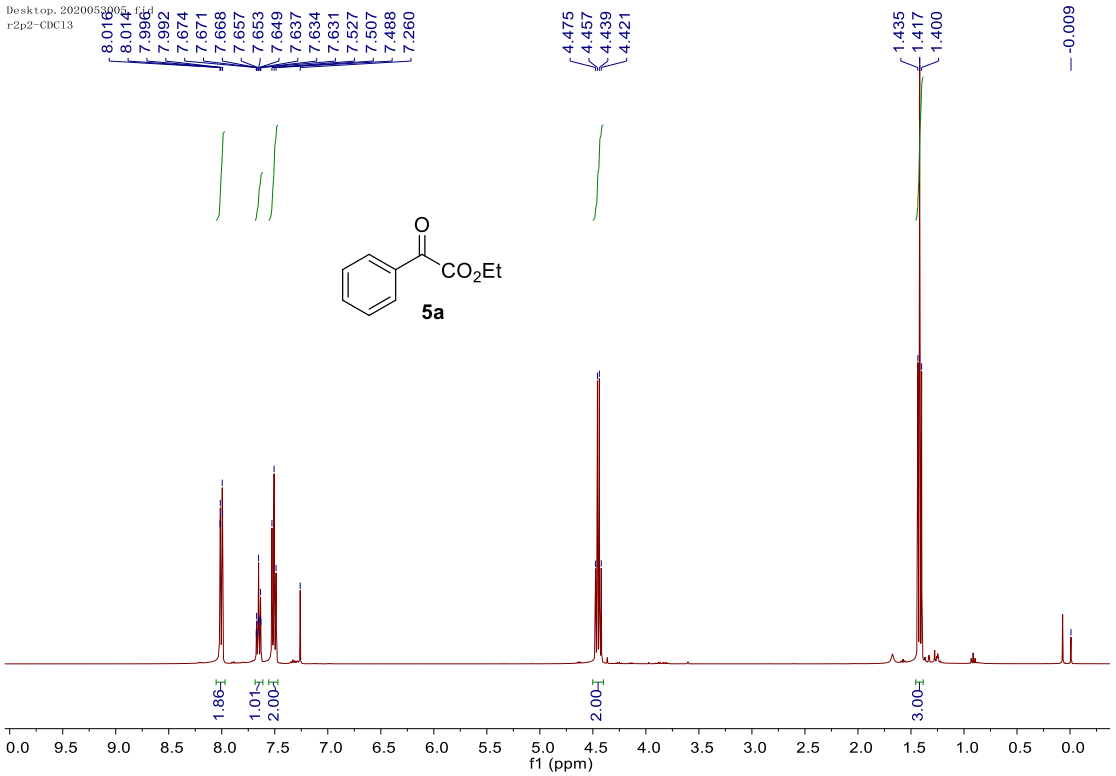


Fig. S23 ^{1}H NMR of compound **5a** in $CDCl_3$.

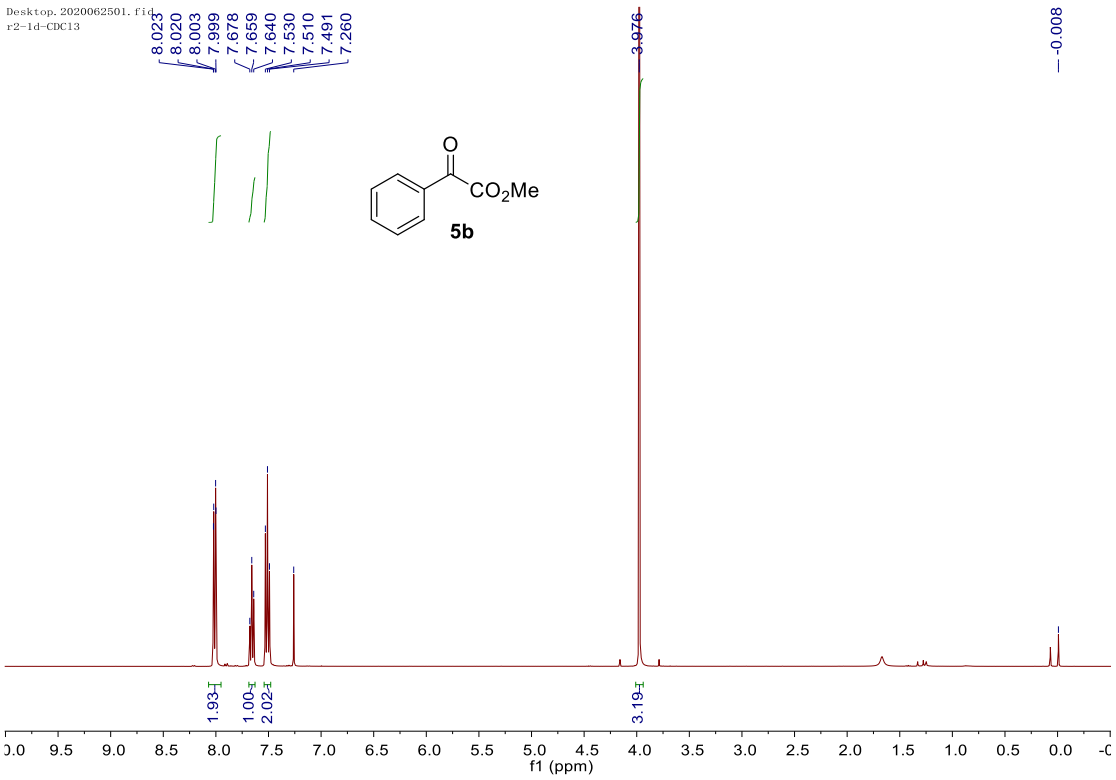


Fig. S24 ^{1}H NMR of compound **5b** in $CDCl_3$.

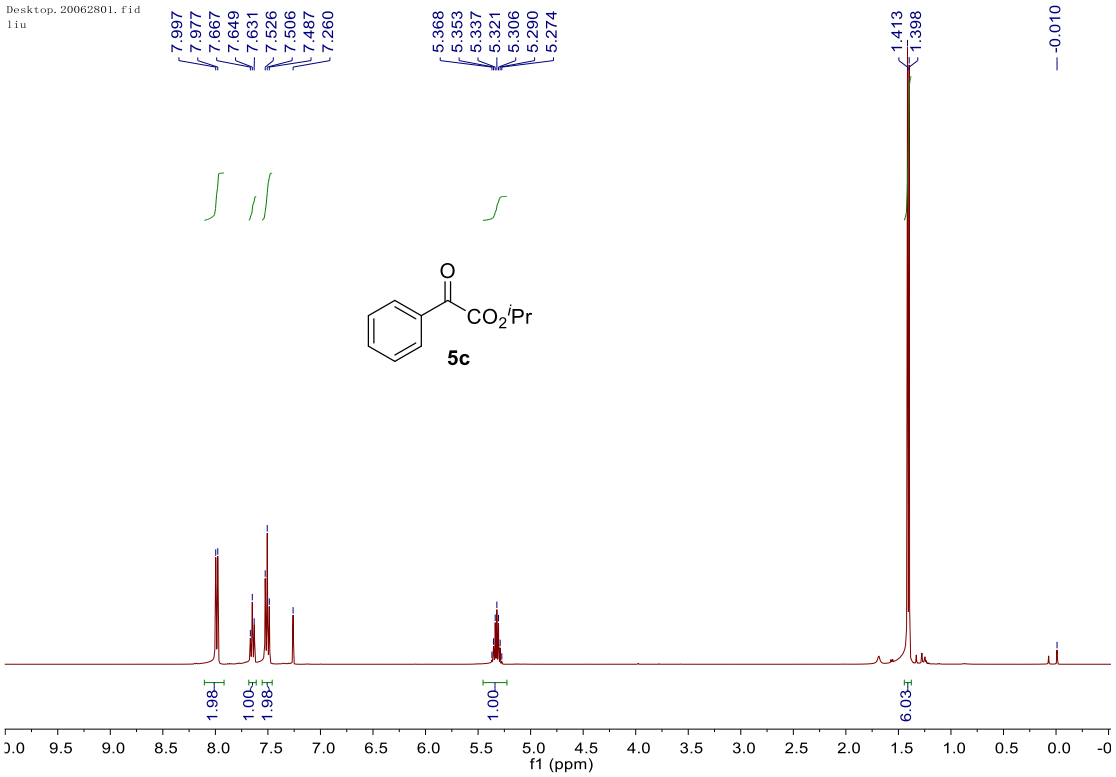


Fig. S25 ^{1}H NMR of compound **5c** in $CDCl_3$.

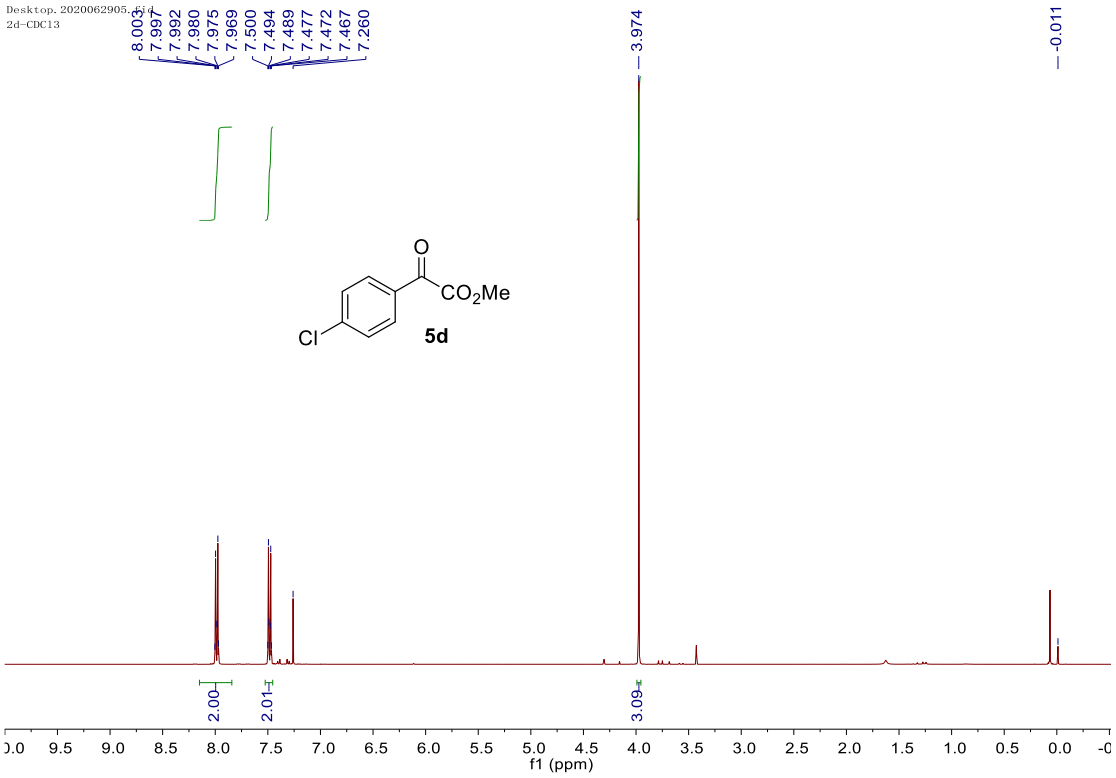


Fig. S26 ^{1}H NMR of compound **5d** in $CDCl_3$.

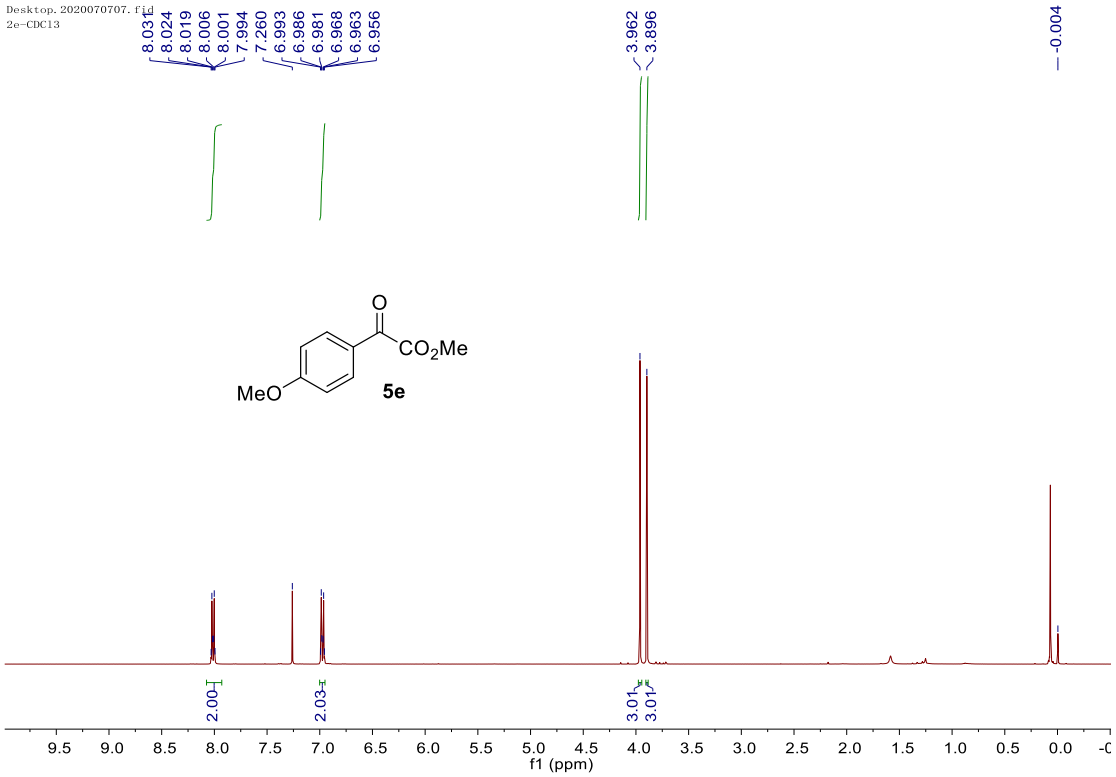


Fig. S27 ^{1}H NMR of compound **5e** in $CDCl_3$.

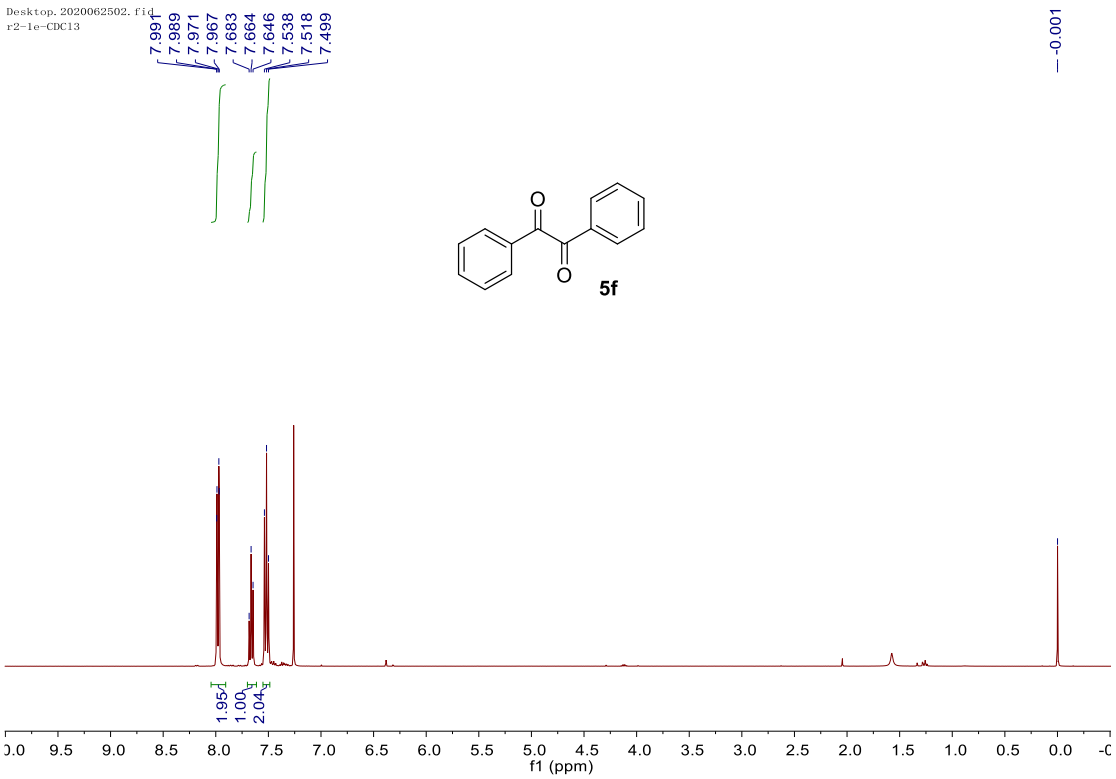


Fig. S28 ^{1}H NMR of compound **5f** in $CDCl_3$.

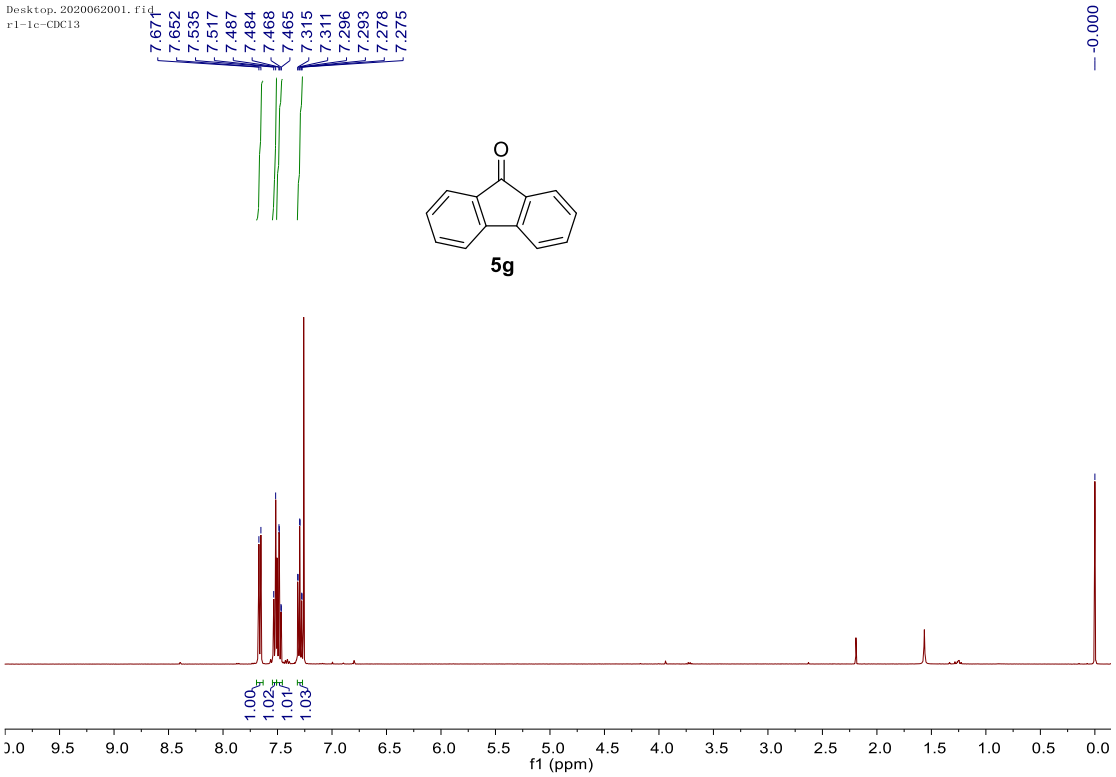


Fig. S29 ^{1}H NMR of compound **5g** in $CDCl_3$.

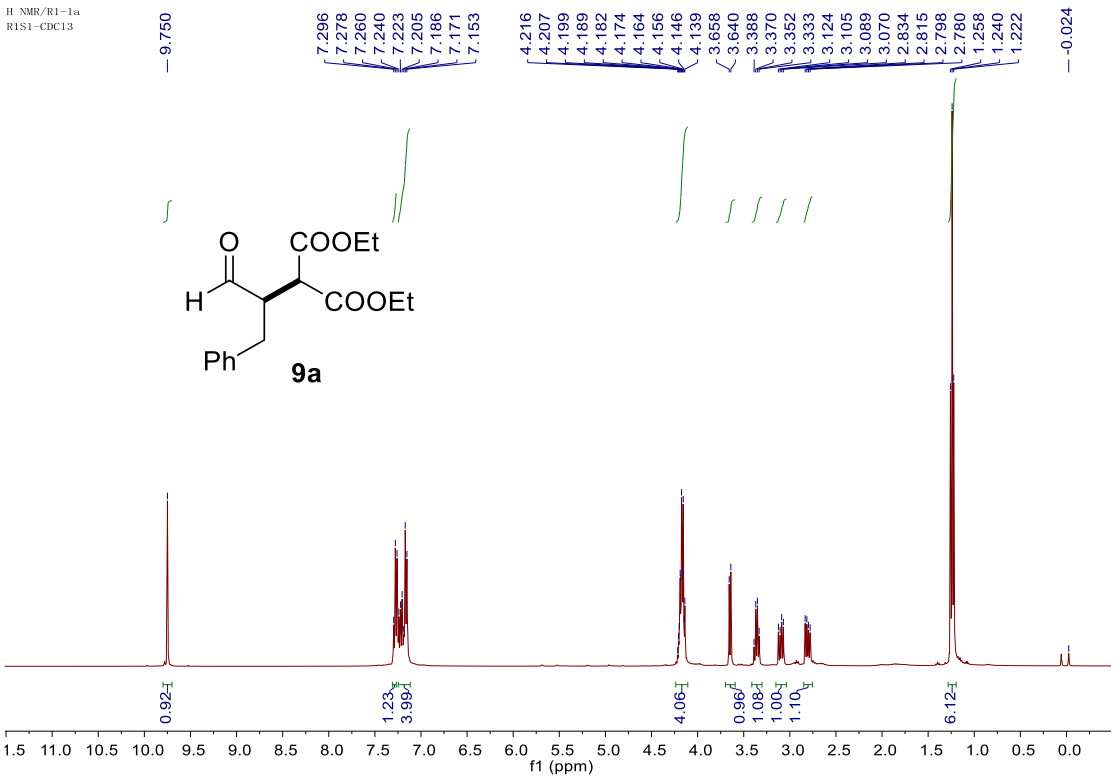


Fig. S30 ^{1}H NMR of compound **9a** in $CDCl_3$.

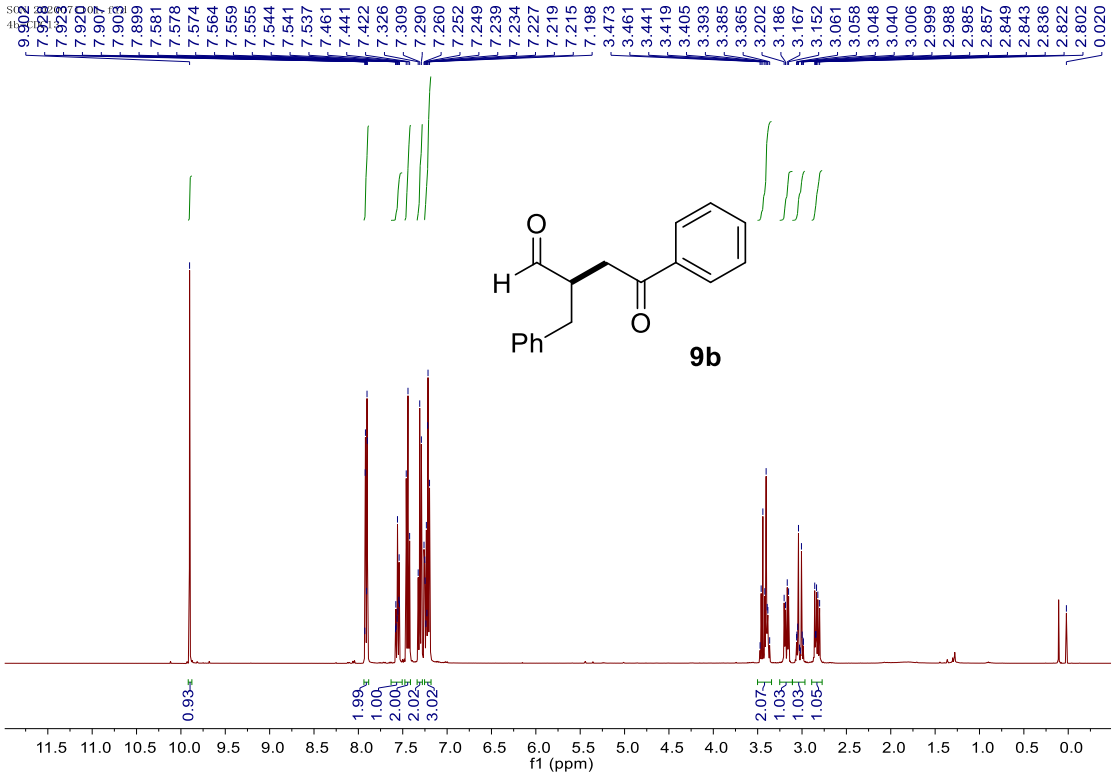


Fig. S31 ^{1}H NMR of compound **9b** in $CDCl_3$.

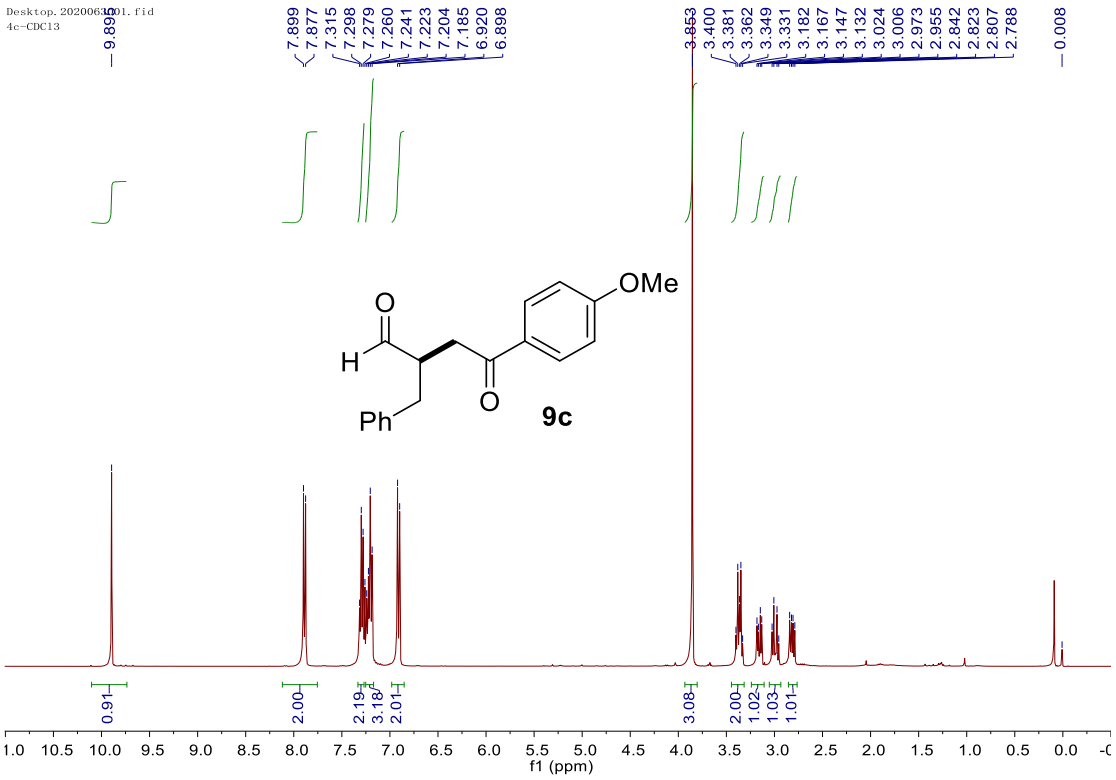


Fig. S32 ^{1}H NMR of compound **9c** in $CDCl_3$.

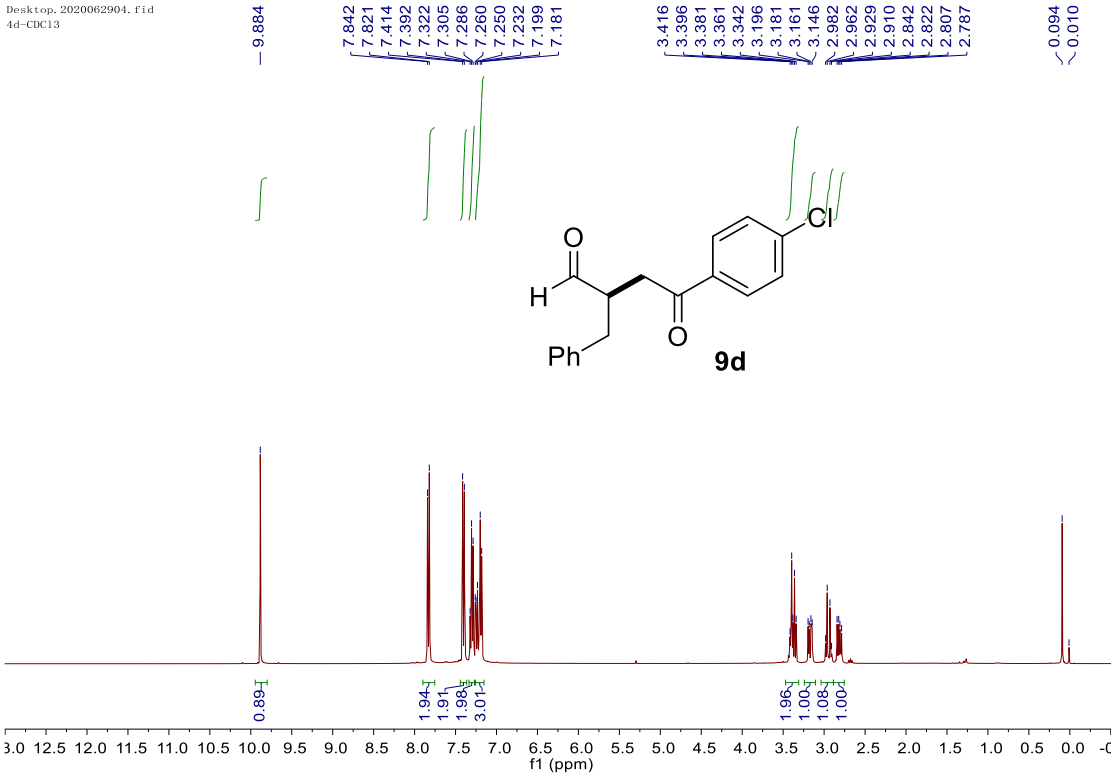


Fig. S33 ^{1}H NMR of compound **9d** in $CDCl_3$.

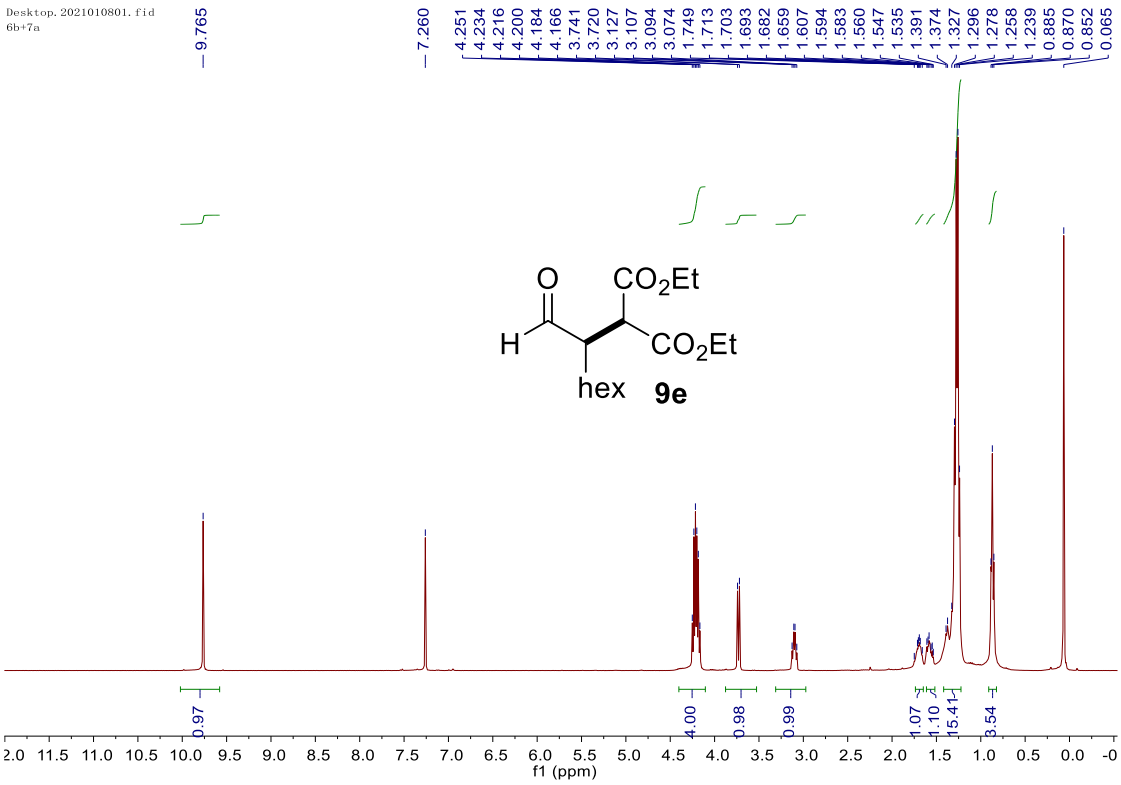


Fig. S34 ^{1}H NMR of compound **9e** in $CDCl_3$.

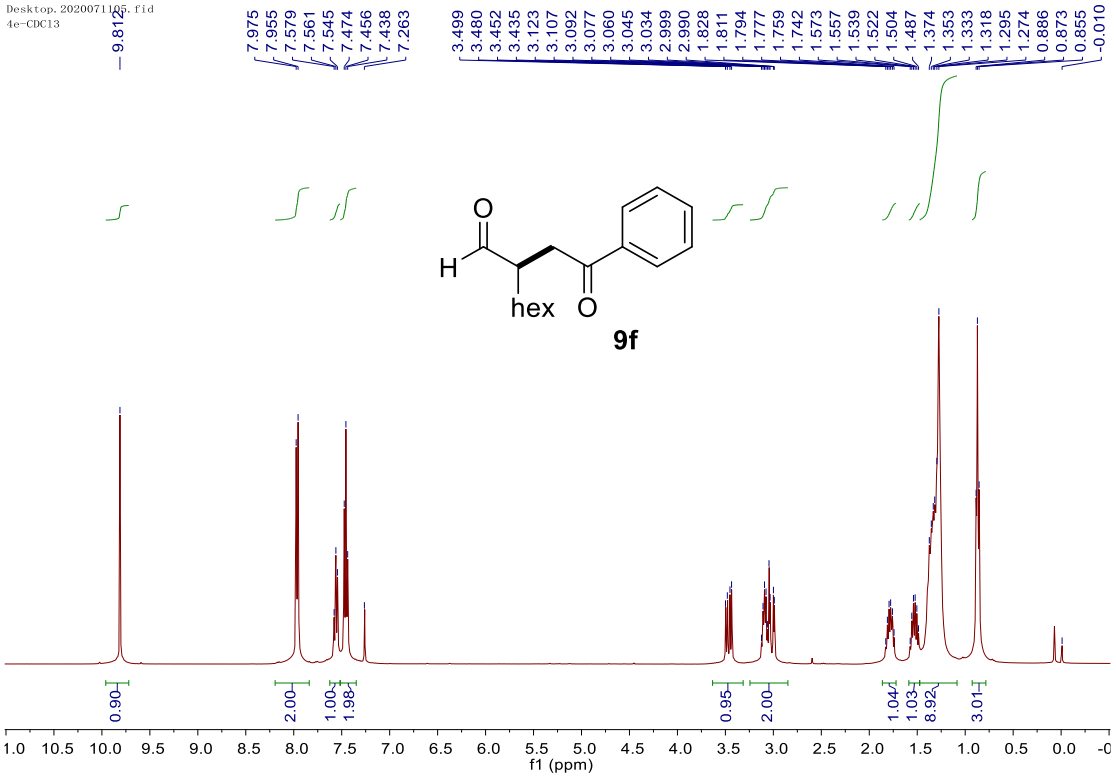


Fig. S35 ^{1}H NMR of compound **9f** in $CDCl_3$.

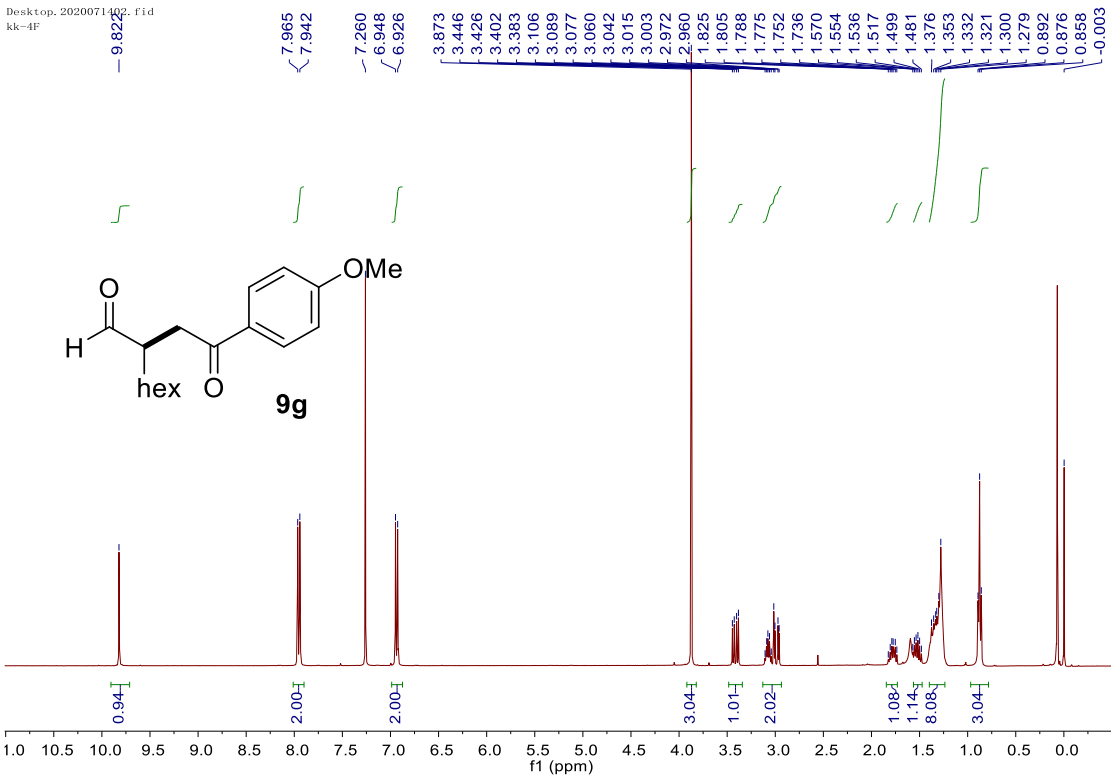


Fig. S36 ^{1}H NMR of compound **9g** in $CDCl_3$.

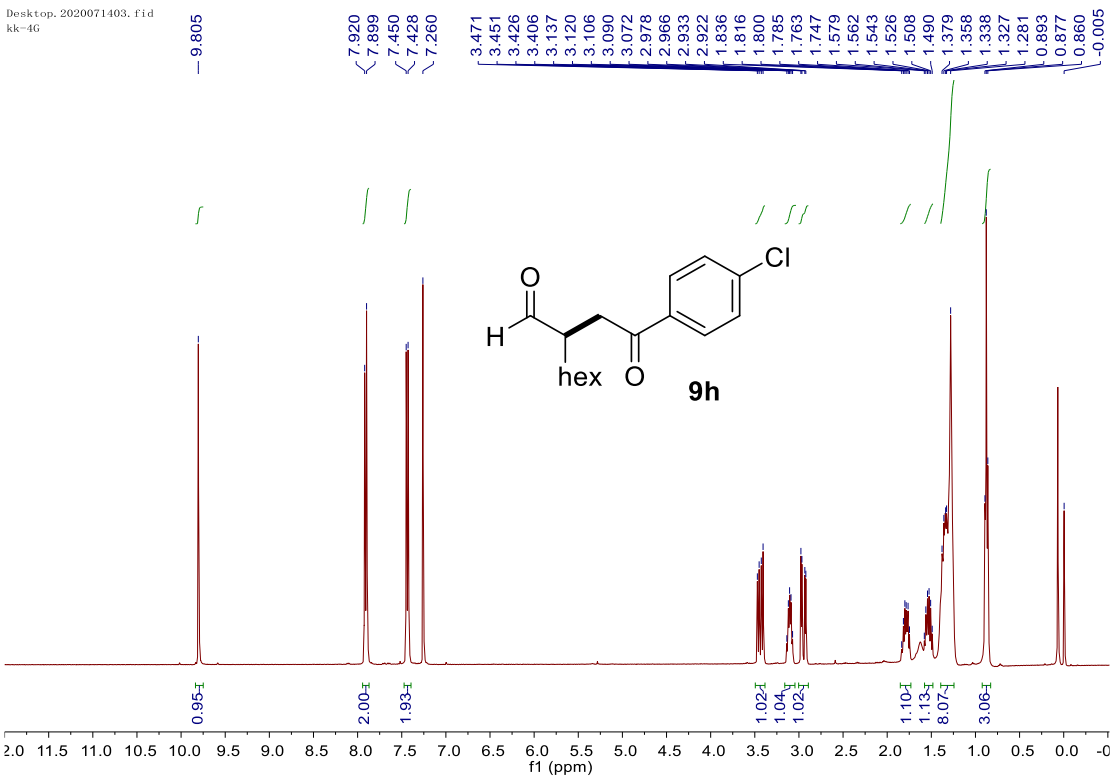
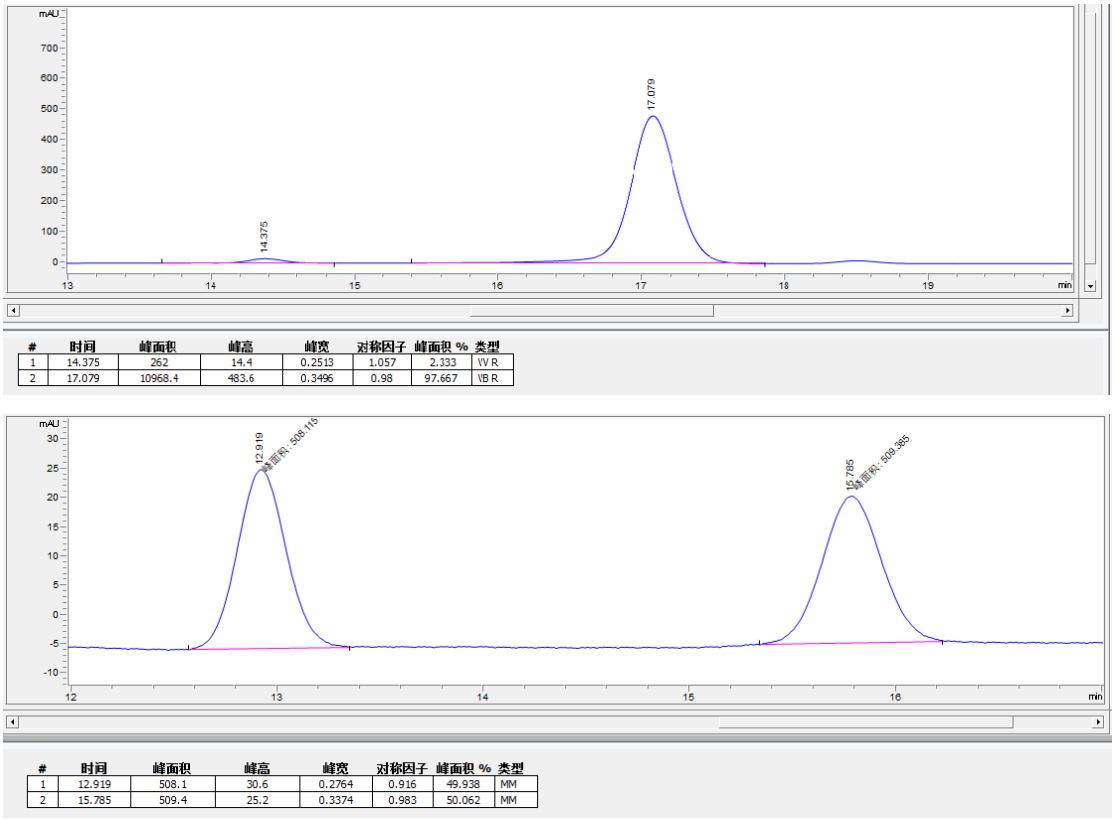


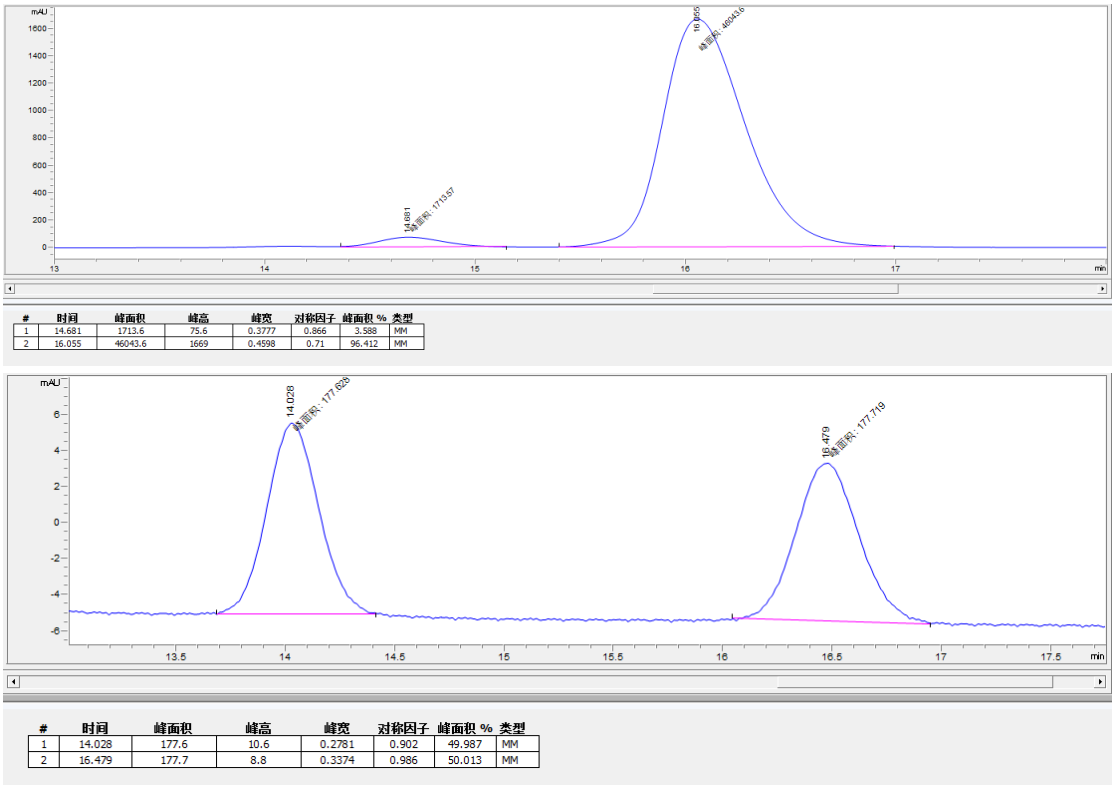
Fig. S37 ^{1}H NMR of compound **9h** in $CDCl_3$.



#	时间	峰面积	峰高	峰宽	对称因子	峰面积 %	类型
1	14.375	262	14.4	0.2513	1.057	2.333	VV R
2	17.079	10968.4	483.6	0.3496	0.98	97.667	VB R

#	时间	峰面积	峰高	峰宽	对称因子	峰面积 %	类型
1	12.919	508.1	30.6	0.2764	0.916	49.938	MM
2	15.785	509.4	25.2	0.3374	0.983	50.062	MM

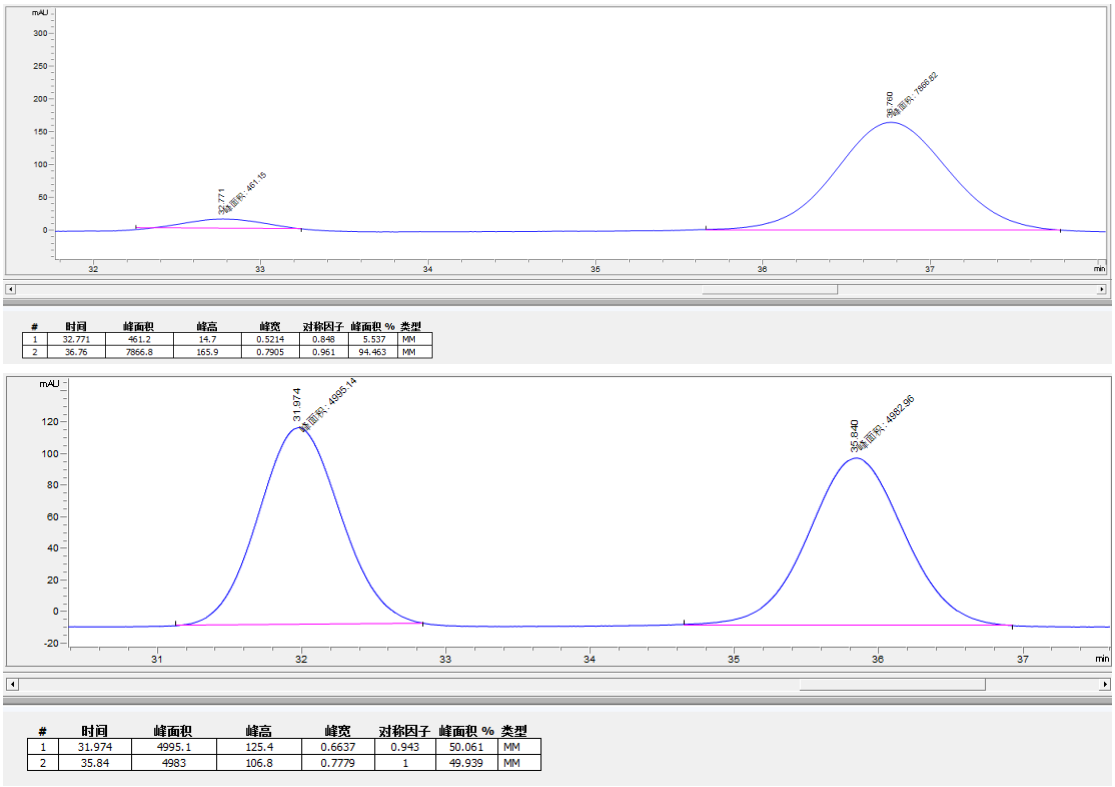
Fig. S38 The enantiomeric excess of **9a** determined by chiral HPLC with IC column (hexane/isopropanol = 90/10; flow 1 mL·min^{-1}).



#	时间	峰面积	峰高	峰宽	对称因子	峰面积 %	类型
1	14.681	1713.6	75.6	0.3777	0.866	3.588	MM
2	16.055	46043.6	1669	0.4598	0.71	96.412	MM

#	时间	峰面积	峰高	峰宽	对称因子	峰面积 %	类型
1	14.028	177.6	10.6	0.2781	0.902	49.987	MM
2	16.479	177.7	8.8	0.3374	0.986	50.013	MM

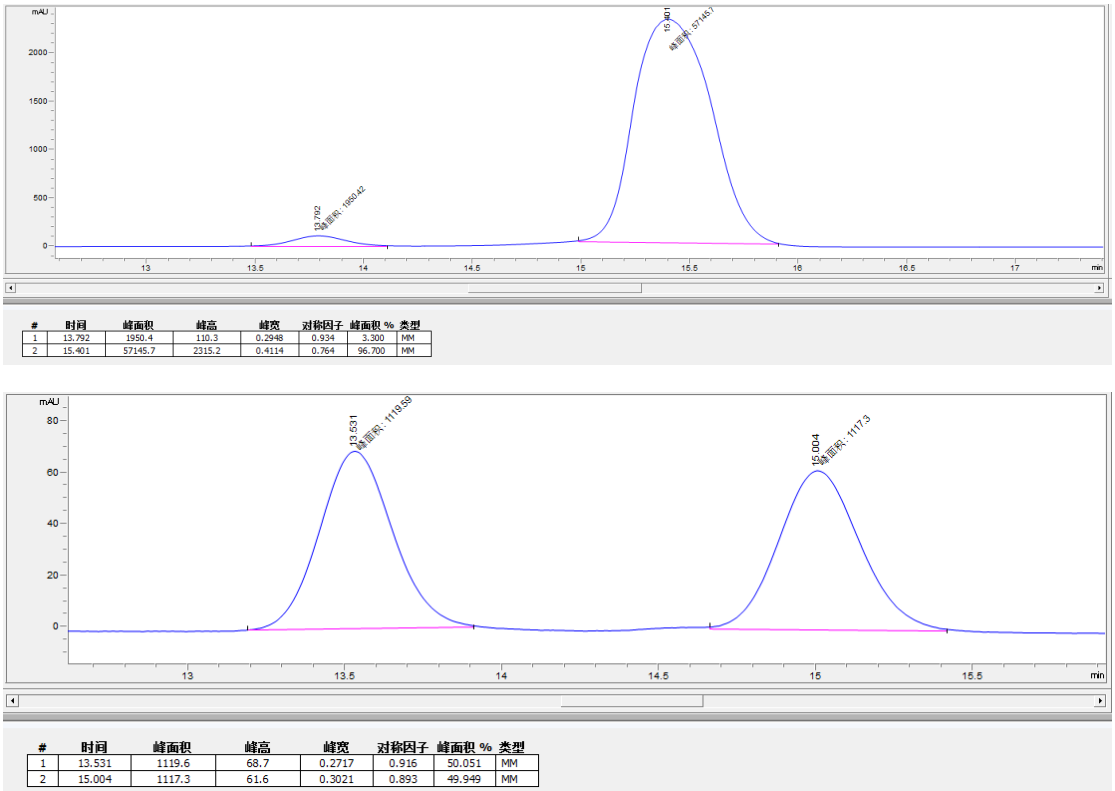
Fig. S39 The enantiomeric excess of **9b** determined by chiral HPLC with IC column (hexane/isopropanol = 95/5; flow 1 mL·min^{-1}).



#	时间	峰面积	峰高	峰宽	对称因子	峰面积 %	类型
1	32.771	461.2	14.7	0.5214	0.848	5.537	MM
2	36.76	7866.8	165.9	0.7905	0.961	94.463	MM

#	时间	峰面积	峰高	峰宽	对称因子	峰面积 %	类型
1	31.974	4995.1	125.4	0.6637	0.943	50.061	MM
2	35.84	4983	106.8	0.7779	1	49.939	MM

Fig. S40 The enantiomeric excess of **9c** determined by chiral HPLC with IC column (hexane/iso38propanol = 90/10; flow 1 mL·min^{-1}).



#	时间	峰面积	峰高	峰宽	对称因子	峰面积 %	类型
1	13.792	1950.4	110.3	0.2948	0.934	3.300	MM
2	15.401	57145.7	2315.2	0.4114	0.764	96.700	MM

#	时间	峰面积	峰高	峰宽	对称因子	峰面积 %	类型
1	13.531	1119.6	68.7	0.2717	0.916	50.051	MM
2	15.004	1117.3	61.6	0.3021	0.893	49.949	MM

Fig. S41 The enantiomeric excess of **9d** determined by chiral HPLC with IC column (hexane/isopropanol = 95/5; flow 1 mL·min^{-1}).

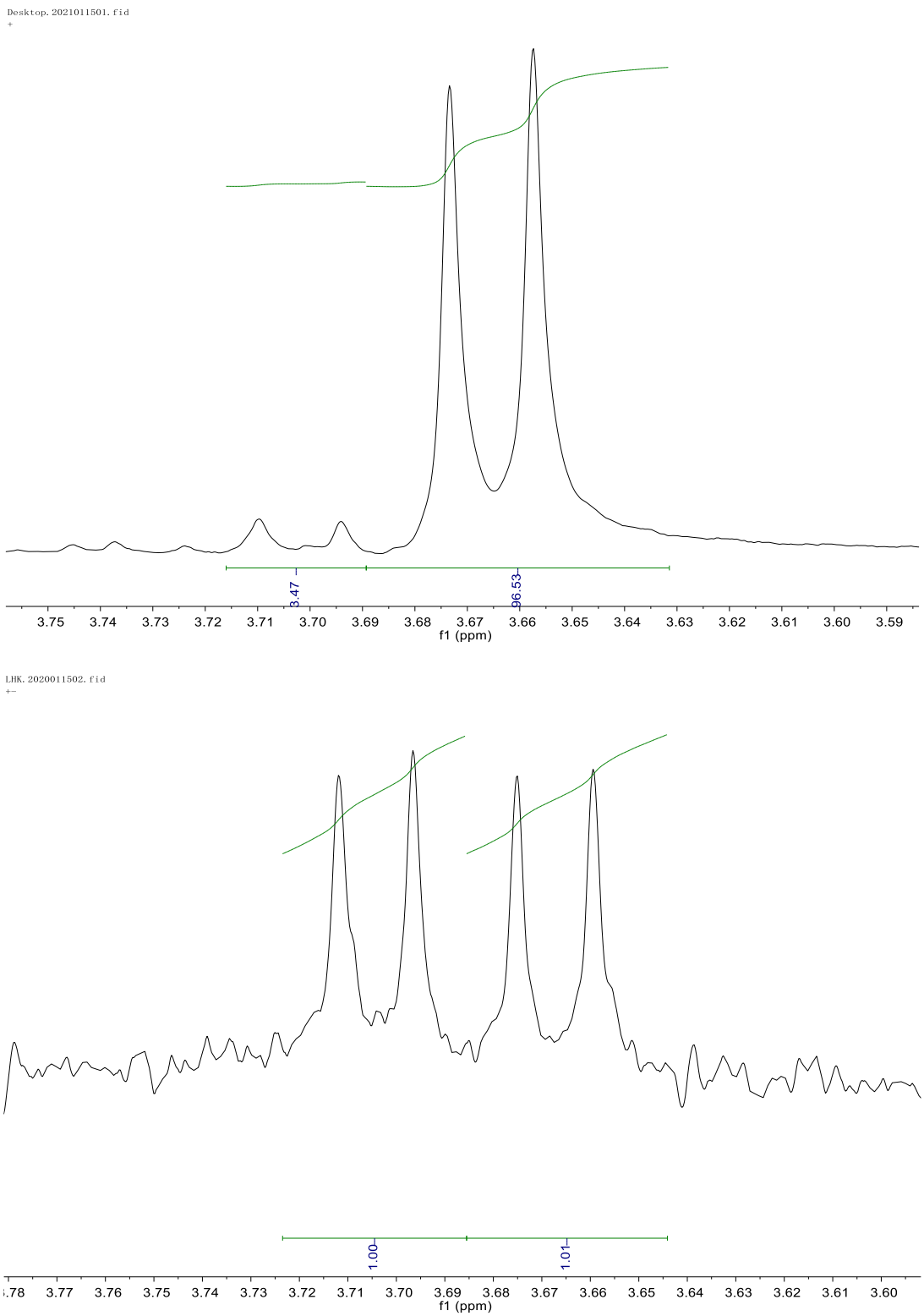
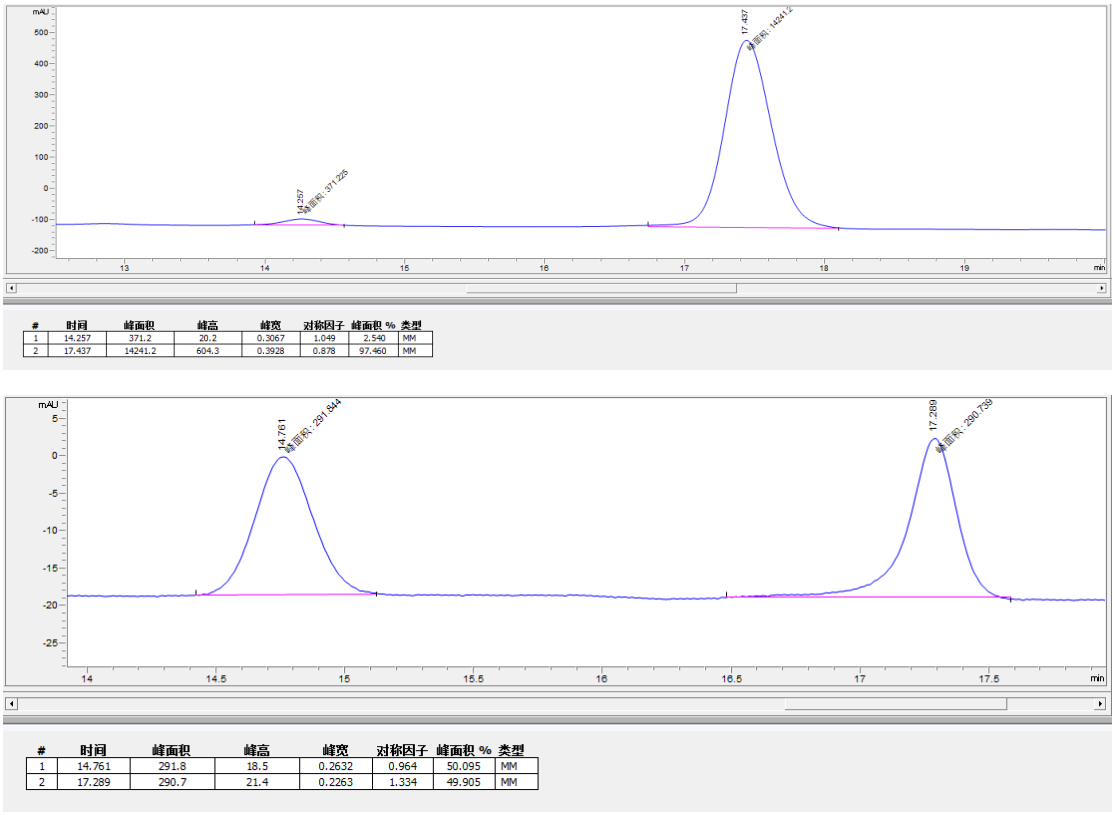


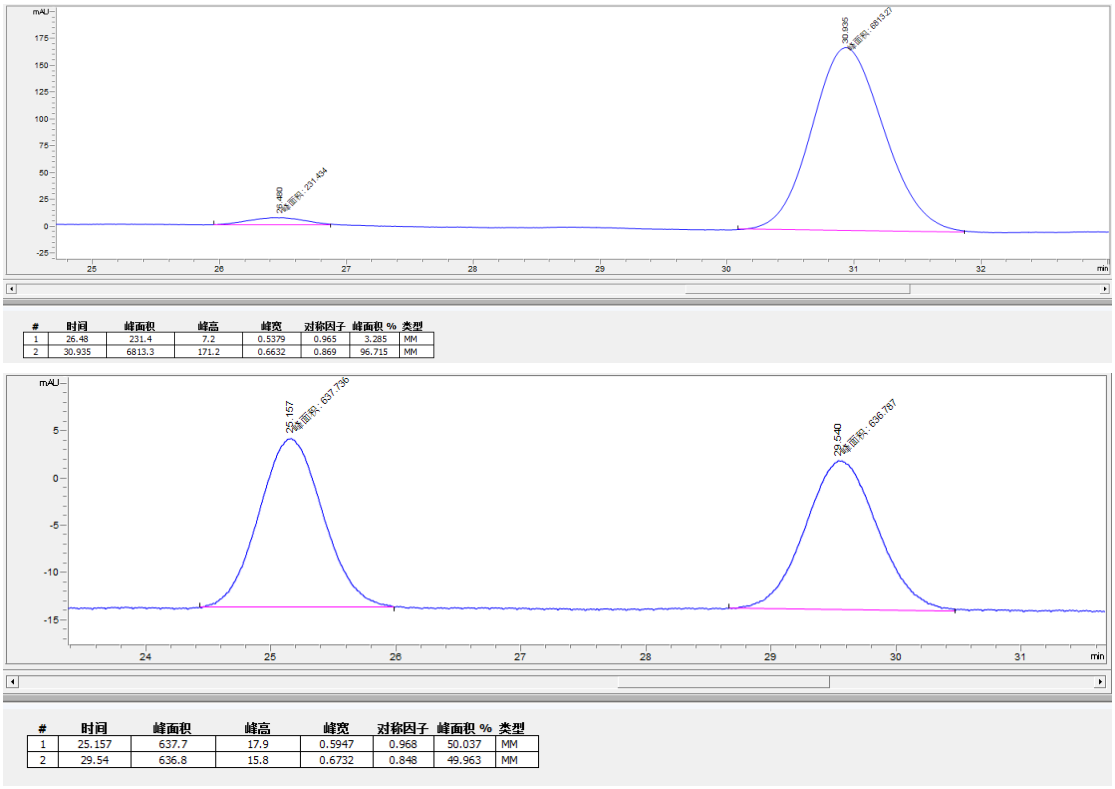
Fig. S42 The enantiomeric excess of **9e** determined by ^{1}H NMR in $CDCl_3$.



#	时间	峰面积	峰高	峰宽	对称因子	峰面积 %	类型
1	14.257	371.2	20.2	0.3067	1.049	2.540	MM
2	17.437	14241.2	604.3	0.3928	0.878	97.460	MM

#	时间	峰面积	峰高	峰宽	对称因子	峰面积 %	类型
1	14.761	291.8	18.5	0.2632	0.964	50.095	MM
2	17.289	290.7	21.4	0.2263	1.334	49.905	MM

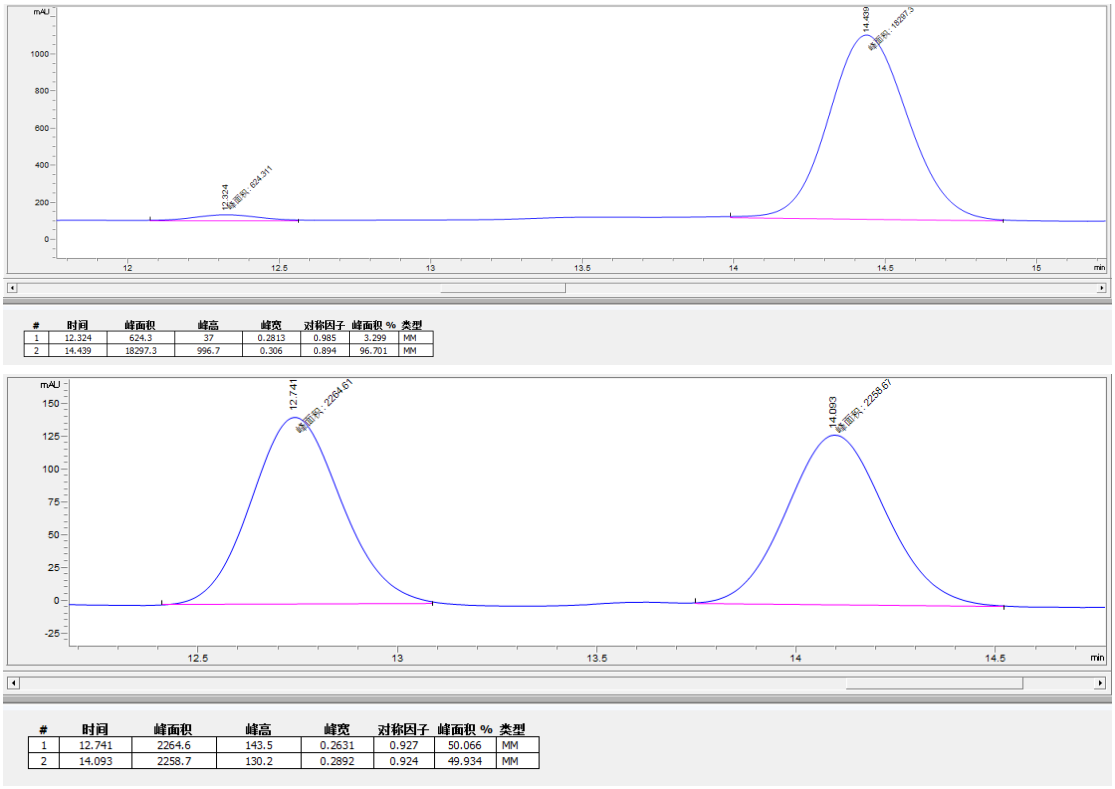
Fig. S43 The enantiomeric excess of **9f** determined by chiral HPLC with IC column (hexane/isopropanol = 95/5; flow 1 mL·min^{-1}).



#	时间	峰面积	峰高	峰宽	对称因子	峰面积 %	类型
1	26.48	231.4	7.2	0.5379	0.965	3.285	MM
2	30.935	6813.3	171.2	0.6632	0.869	96.715	MM

#	时间	峰面积	峰高	峰宽	对称因子	峰面积 %	类型
1	25.157	637.7	17.9	0.5947	0.968	50.037	MM
2	29.54	636.8	15.8	0.6732	0.848	49.963	MM

Fig. S44 The enantiomeric excess of **9g** determined by chiral HPLC with IC column (hexane/isopropanol = 90/10; flow 1 mL·min^{-1}).



#	时间	峰面积	峰高	峰宽	对称因子	峰面积 %	类型
1	12.324	624.3	37	0.2813	0.985	3.299	MM
2	14.439	18297.3	996.7	0.306	0.894	96.701	MM

#	时间	峰面积	峰高	峰宽	对称因子	峰面积 %	类型
1	12.741	2264.6	143.5	0.2631	0.927	50.066	MM
2	14.093	2258.7	130.2	0.2892	0.924	49.934	MM

Fig. S45 The enantiomeric excess of **9h** determined by chiral HPLC with IC column (hexane/isopropanol = 95/5; flow 1 mL·min^{-1}).