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Supporting Information

A powerful azomethine ylide route mediated by TiO_2 photocatalysis for preparation of polysubstituted imidazolidines

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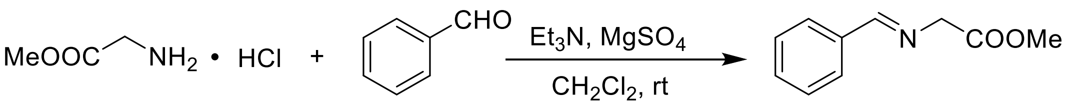
I. General Information

All reactions were performed under Ar atmospheres in oven-dried glassware with magnetic stirring. Unless otherwise stated, all reagents were purchased from commercial suppliers and used without further purification. Photochemical reactions were carried out using a HX-CFL 300W Xe lamp purchased from Au Light (Beijing) using standard Pyrex-glass bottles with sealable rubber-cap. GC and GC-MS were performed on an Agilent 7890 A equipped with a split-mode capillary injection system and Agilent 5973 network mass spec detector (MSD). Yield refers to isolated yield of analytically pure material unless otherwise noted. Thin layer chromatography (TLC) was performed on SilicaPlate 250 μm thick silica gel plates provided by Qingdao chemicals. Column chromatography was performed using Qingdao Haiyang flash silica gel (200-300 mesh). Visualization was accomplished with short wave UV light (254 nm). ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$, CD_3CN or $(CD_3)_2CO$ using 300 MHz, 400 MHz or 600 MHz Bruker-Avance instruments. Chemical shifts (δ, ppm) are relative to tetramethylsilane (TMS) with the resonance of the non-deuterated solvent or TMS as the internal standard. ^{1}H NMR data are reported as follows: chemical shift, multiplicity (s = singlet; d = doublet; q = quartet; m = multiplet), coupling constant (Hz), and integral. Data for ^{13}C NMR spectra are reported in terms of chemical shift. Accurate mass measurements were performed using a SolariX mass spectrometer with electrospray ionization in positive mode. Single-crystal X-ray diffraction data were collected on a Supernova Dual Source diffractometer, equipped with an Atlas detector using Cu Kα radiation (λ= 1.54178 Å). The CrysAlisPro software system was used for the data collection, cell refinement, and data reduction.

II. General procedures for the preparation of α-iminoester



α-Iminoester was prepared using the reported procedure. A suspension of methyl glycinate hydrochloride (14.8 mmol), $MgSO_4$ (14.8 mmol) and Et_3N (14.8 mmol) in dry CH_2Cl_2 (36 mL) was stirred at room temperature for 1 h, and benzaldehyde (9.9 mmol) was added. After 12 h at room temperature, the mixture was filtered off and water (5 mL) was added. The organic layer was separated and the aqueous phase was extracted with CH_2Cl_2 (10 mL). The combined organic layers were washed with brine, dried over $MgSO_4$, and evaporated under reduced pressure to afford α-iminoester which was used in the next step without further purification.

III. General procedures A for TiO_2 photocatalyzed 1,3-dipolar cycloaddition of N-benzylidinebenzylamine and aliphatic alcohol

A 10 mL glass vial filled with 3 mL extra-dry aliphatic alcohol, 1 mmol N-benzylidinebenzylamine (NBBA), 32 mg P25-TiO_2 photocatalyst was capped and magnetically stirred in front of a 300 W Xe-lamp ($\lambda > 365$ nm). A high power axial fan was placed beside the glass vial to keep the reaction under room temperature. Once the starting material was completely consumed (monitored by TLC, petroleum ether/EtOAc=10/1), the mixture was concentrated to dryness. The residue was purified through column chromatography (petroleum ether/EtOAc =10/1-200/1) to afford the corresponding cycloaddition product. The product was analyzed by HR-ESI-MS, ^{1}H NMR, and ^{13}C NMR.

IV. General procedures B for TiO_2 photocatalyzed 1,3-dipolar cycloaddition of N-benzylidinebenzylamine and aromatic alcohol

A 10 mL glass vial filled with 3 mL extra-dry acetonitrile, 3 mmol aromatic alcohol, 1 mmol N-benzylidinebenzylamine, 32 mg P25-TiO_2 photocatalyst was capped and magnetically stirred in front of a 300 W Xe-lamp ($\lambda > 365$ nm). A high-power axial fan was placed beside the glass vial to keep the reaction under room temperature. Once the starting material was completely consumed (monitored by TLC, petroleum ether/EtOAc=10/1), the mixture was concentrated to dryness. The residue was purified through column chromatography (petroleum ether/EtOAc =10/1-200/1) to afford the corresponding cycloaddition product. The product was analyzed by HR-ESI-MS, ^{1}H NMR, and ^{13}C NMR.

V. Photocatalyst reuse experiments for 5 cycles

1. Photocatalyst performance for 5 cycles

For the photocatalyst stability test: after one standard reaction, the photocatalyst was separated by centrifugation, and washed by acetonitrile twice, and then dispersed in 2 mL aliphatic alcohol or acetonitrile and transferred into the photo-reactor, the following steps are the same to the procedure in the above-mentioned standard reaction condition. The cycle stability test was repeated for five times. The conversion and yield were shown in **Figure S1**.

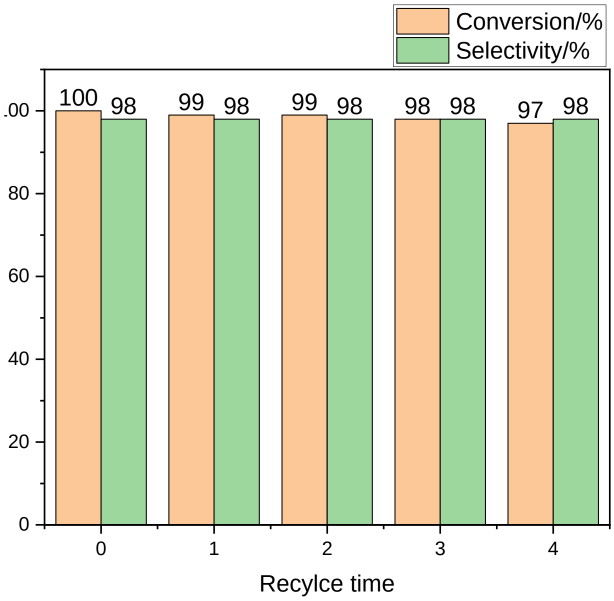


Figure S1 Recycling of P25 TiO_2 photocatalyst in the 1,3-dipolar cycloaddition between ethanol and NBBA Reaction conditions: NBBA (1 mmol), ethanol (3 mL), P25 TiO_2 32 mg, Ar atmosphere, 300 W Xe lamp irradiation for 5 h at 5 cm.

TEM Characterization:

The morphologies of the fresh and recycled P25 TiO_2 samples were investigated by microscopic technique. Powders of catalyst samples were dispersed in ethanol and drop-casted on Cu-grids. HAADF-STEM analysis was conducted on JEOL 200F transmission electron microscope with a 300 kV accelerating voltage.

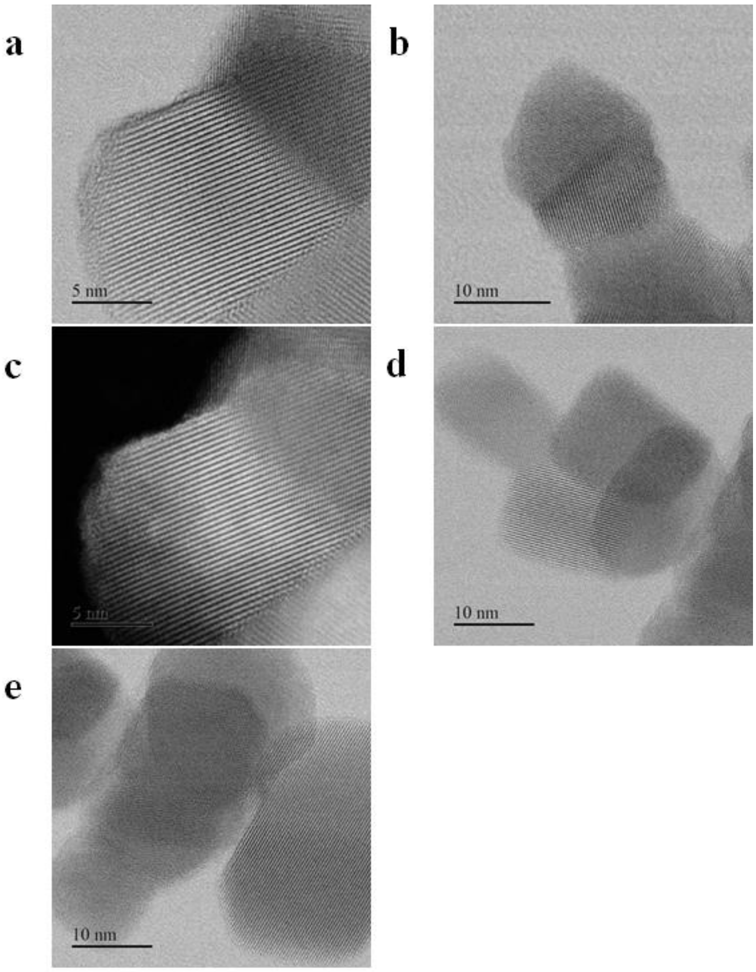


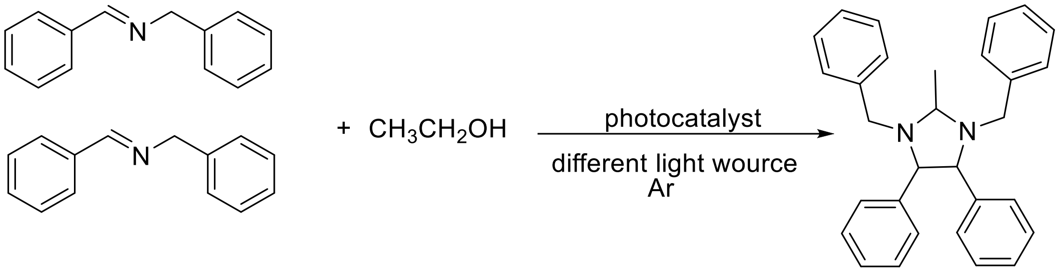
Fig. S2. Fresh and recycled TiO_2 photocatalyst HAADF-STEM images (**a**) HAADF-STEM image of fresh unused P25 TiO_2 sample indicates the lattice fringes corresponding to (101) crystal plane of anatase component, (110) crystal plane of rutile component. (**b**) HAADF-STEM image of P25 TiO_2 samples after 1 time recycle. (**c**) HAADF-STEM image of P25 TiO_2 samples after 2 times recycle.(**d**) HAADF-STEM image of P25 TiO_2 samples after 3 times recycle.(**e**), HAADF-STEM image of P25 TiO_2 samples after 4 times recycle.

VI. TiO_2 photocatalyzed 1,3-dipolar cycloaddition of benzylidinebenzyl amine and alcohols on gram scale

Under Ar atmosphere N-benzylidinebenzylamine (1.482 g, 7.6 mmol) and P25-TiO_2 (0.08g, 1 mmol) were dissolved in 10 mL of ethanol and stirred at room temperature and irradiated with a 300 W Xe-lamp for 48 h. And then was purified through column chromatography above to give the product 1a in 91 % yield (1.45 g).

VII. Optimization of reaction conditions

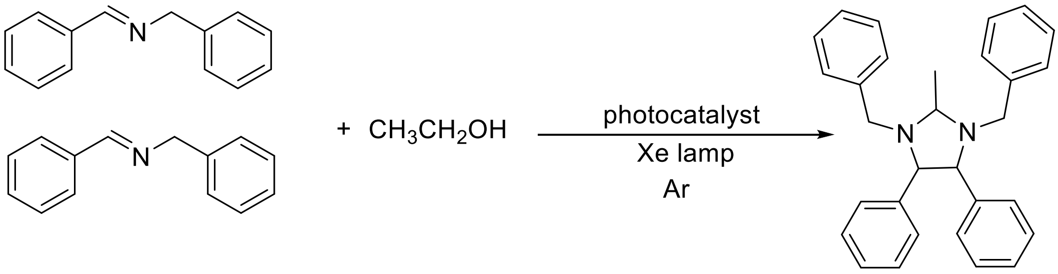
Table S1. TiO_2 photocatalytic Imidazolidine ring formation using different light source



Entry	Light source	Yield[a] (%)
1	300 W xenon lamp with 365 nm filter	98
2	300 W xenon lamp with 420 cut-off filter	15
3	100 W 395 nm LED	80
4	100 W 455 nm LED	trace

Reaction conditions: 1 mmol N-benzylidinebenzylamine, ethanol 3 ml as solvent， P25 TiO_2 32 mg, Ar atmosphere under different light source illumination for 5 h at 5 cm distance. [a] Measured by GC.

Table S2. Optimization of photocatalysts for photocatalytic imidazolidine formation from ethanol and N-benzylidinebenzylamine



Entry	Photocatalyst	Light Source	Time	Yield[a]

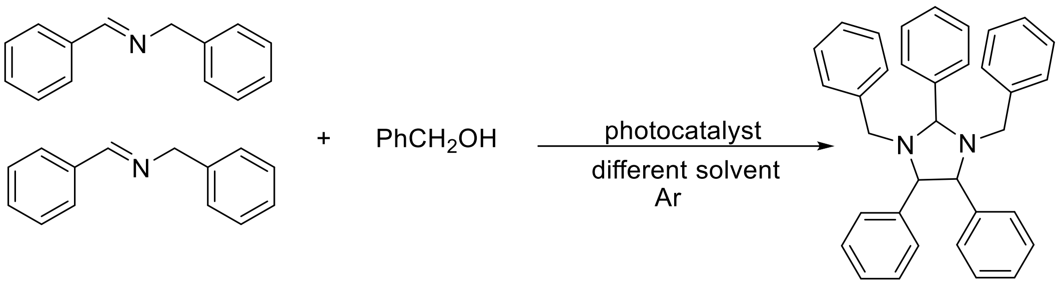
1	TiO_2 (Aeroxide P25)	Xe lamp	5 h	98%
2	TiO_2 anatase	Xe lamp	5 h	95%
3	TiO_2 rutile	Xe lamp	5 h	38%
4	CdS	Xe lamp with 420 nm filter	8 h	trace
5	mpg-C_3N_4[b]	Xe lamp with 420 nm filter	24 h	n.d.
6	Eosin Y	Xe lamp with 420 nm filter	24 h	n.d.
7	Ru(bpy)$_3$Cl$_2$[c]	Xe lamp with 420 nm filter	24 h	n.d.
8	Ir(ppy)$_3$[c]	Xe lamp with 420 nm filter	24 h	n.d.
9	$BiVO_4$	Xe lamp with 420 nm filter	10 h	n.d.
10	Perylene bis(diimide)	Xe lamp with 420 nm filter	24 h	n.d.

Reaction conditions:1 mmol N-benzylidinebenzylamine, ethanol 3 ml as solvent，photocatalyst 0.4 eq, Ar atmosphere under illumination for a given time at 5 cm distance.

[a]Isolated yield after column chromatography. [b]37 mg mpg-C_3N_4 photocatalyst was used.

[c]0.05 eq photocatalyst was used.

Table S3. TiO_2 photocatalytic Imidazolidine ring formation using different solvent for benzyl alcohol and N-benzylidinebenzylamine

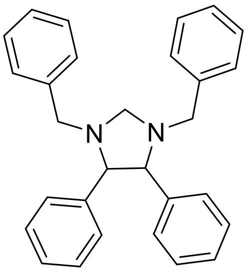


Entry	Solvent	Yield[a] (%)
1	ethanol	98
2	MeCN	75
3	DMF	45
4	DMSO	38
5	n-hexane	12
6	toluene	18
7	H_2O	trace

Reaction conditions: 1 mmol N-benzylidinebenzylamine, 3 ml solvent, P25 TiO_2 32 mg, Ar atmosphere under 300 W xenon lamp illumination for 5 h at 5 cm distance. [a]Measured by GC.

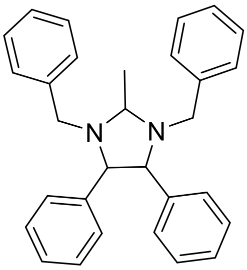
VIII. Characterization data for TiO_2 photocatalyzed 1,3-dipolar cycloaddition of N-benzylidinebenzylamine and alcohol products

1a 1,3-dibenzyl-4,5-diphenylimidazolidine



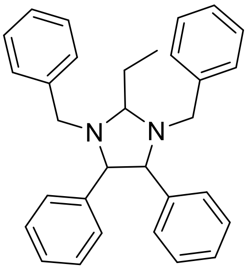
Prepared according to the general procedure A as described above in 98% yield. (monitored by TLC, petroleum ether:EtOAc=30:1, $R_f = 0.40$) It was purified by column chromatography (PE/EtOAc=100/1) to afford white solid. ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.40-7.08 (m, 20H), 4.26 (s, 1H), 4.24 (d, 4.2 Hz, 0.65H), 3.99 (d, 13.6 Hz, 1H), 3.95 (d, 13.6 Hz, 0.66H), 3.88 (s, 0.64H), 3.80 (s, 0.65H), 3.50 (d, 13.6 Hz, 2H), 3.27 (d, 4.2 Hz, 0.61H); ^{13}C NMR (151 MHz, Chloroform-*d*) δ 139.33, 139.06, 139.01, 138.49, 128.44, 128.00, 127.72, 127.62, 127.00, 126.89, 126.41, 126.32, 126.12, 77.01, 75.41, 74.17, 71.92, 57.14, 56.49; HR-ESI-MS: m/z (ESI) calcd for $C_{29}H_{29}N_2$ $(M+H)^+$ 405.23252, found 405.23247

1b 1,3-dibenzyl-2-methyl-4,5-diphenylimidazolidine



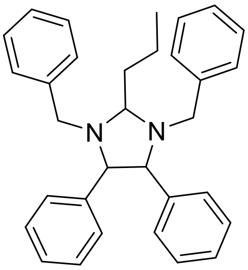
Prepared according to the general procedure A as described above in 98% yield. (monitored by TLC, petroleum ether:EtOAc=30:1, $R_f = 0.38$) It was purified by column chromatography (PE/EtOAc=100/1) to afford white solid. ^{1}H NMR (600 MHz, Acetone-*d₆*) δ 7.41 (d, 7.7 Hz, 2H), 7.32-7.16 (m, 18H), 4.21 (q, 5.8 Hz, 1H), 3.96 (d, 10.4 Hz, 1H), 3.88 (s, 1H), 3.87 (d, 6.2 Hz, 1H), 3.72(d, 13.4 Hz, 1H), 3.59 (d, 14.7 Hz, 1H), 3.52 (d, 14.7 Hz, 1H), 0.98 (d, 5.8 Hz, 3H); ^{13}C NMR (151 MHz, Acetone-*d₆*) δ 140.82, 139.76, 139.55, 139.40, 129.03, 128.36, 128.25, 128.18, 127.98, 127.94, 127.82, 127.74, 127.64, 127.23, 126.83, 126.59, 77.19, 75.06, 73.74, 57.96, 49.74, 16.93; HR-ESI-MS: m/z (ESI) calcd for $C_{30}H_{31}N_2$ $(M+H)^+$ 419.24818, found 419.24789

1c 1,3-dibenzyl-2-ethyl-4,5-diphenylimidazolidine



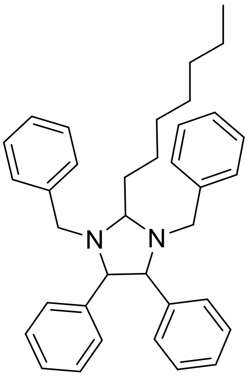
Prepared according to the general procedure A as described above in 90% yield. (monitored by TLC, petroleum ether:EtOAc=30:1, $R_f = 0.38$) It was purified by column chromatography (PE/EtOAc=250/1) to afford white oil. ^{1}H NMR (600 MHz, Acetone-*d₆*) δ 7.45-6.90 (m, 20H), 4.27 (q, 6.9 Hz, 1H), 4.10 (t, 7.0 Hz, 0.41 H), 4.05 (d, 3.6 Hz, 0.43H), 4.01 (d, 14.3 Hz, 0.52H), 3.93 (d, 14.9 Hz, 0.61H), 3.85 (q, 13.9 Hz, 0.36 H), 3.79 (d, 13.7 Hz, 0.47 H), 3.71 (d, 13.9 Hz, 0.62H), 3.65 (d, 14.7 Hz, 0.46H), 3.54 (t, 7.4 Hz, 0.52H), 3.43 (dd, 13.7Hz, 1H), 1.32 (m, 2H), 1.10 (d, 6.7 Hz, 1H), 0.92 (d, 6.9 Hz, 2H); ^{13}C NMR (151 MHz,Acetonitrile-d_3) δ 141.17, 139.91, 139.50, 139.12, 138.24, 129.76, 129.72, 129.52, 129.07, 128.55, 128.37, 128.22, 128.17, 128.11, 128.04, 128.01, 127.90, 127.86, 127.77, 127.71, 127.64, 127.39, 126.96, 126.89, 126.53, 126.30, 83.36, 82.91, 77.32, 73.96, 73.13, 58.36, 57.14, 50.11, 22.40, 13.39; HR-ESI-MS: m/z (ESI) calcd for $C_{31}H_{33}N_2$ (M+H)$^+$ 433.26383, found 433.26389

1d 1,3-dibenzyl-4,5-diphenyl-2-propylimidazolidine



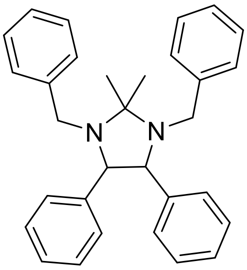
Prepared according to the general procedure A as described above in 82% yield. (monitored by TLC, petroleum ether:EtOAc=30:1, R_f = 0.37) It was purified by column chromatography (PE/EtOAc=250/1) to afford white oil. ^{1}H NMR (600 MHz, Acetone-d_6) δ 7.40-6.93 (m, 20H), 4.07 (s, 0.24H), 4.04 (d, 3.0 Hz, 0.36H), 4.23 (s, 0.24H), 4.01 (d, 8.6 Hz, 1H), 3.93 (d, 4.5 Hz, 0.74H), 3.90 (s, 0.86H), 3.80 (t, 13.7 Hz, 1.24H), 3.67 (d, 14.1 Hz, 0.42H), 3.54 (s, 2H), 1.56-1.42 (m, 2H), 0.91 (t, 7.0 Hz, 2H), 0.76 (t, 6.9 Hz, 3H); ^{13}C NMR (151 MHz,Acetonitrile-d_3) δ 142.83, 141.29, 140.46, 140.20, 130.35, 130.13, 130.00, 129.40, 129.23, 129.07, 128.84, 128.81, 128.58, 127.93, 127.75, 127.56, 79.77, 77.45, 75.10, 59.94, 50.43, 33.24, 19.09, 14.47; HR-ESI-MS: m/z (ESI) calcd for $C_{32}H_{35}N_2$ (M+H)$^+$ 447.27948, found 447.27920

1e 1,3-dibenzyl-2-heptyl-4,5-diphenylimidazolidine



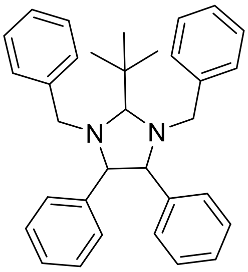
Prepared according to the general procedure A as described above in 60% yield. (monitored by TLC, petroleum ether:EtOAc=30:1, $R_f = 0.39$) It was purified by column chromatography (PE/EtOAc=150/1) to afford white solid. ^{1}H NMR (400 MHz, Acetonitrile-*d$_3$*) δ 7.40-6.92 (m, 20H), 4.09 (q, 7.3 Hz, 1H), 4.08 (s, 0.5H), 4.01 (dd, 8.1 Hz, 1H), 3.91 (dd, 10.2 Hz, 1H), 3.82 (d, 13.8 Hz, 0.5H), 3.77 (d, 13.9 Hz, 0.8H), 3.66 (d, 14.6Hz, 1H), 3.51 (d, 14.8 Hz, 1.6H), 1.71 (d, 4.5 Hz, 2H), 1.55-1.39 (m, 2H), 1.27-1.15(m, 8H), 0.93 (t, 8.1 Hz, 3H); ^{13}C NMR (100 MHz,Acetonitrile-*d$_3$*) δ 141.90, 140.82, 140.67, 140.40, 138.04, 130.66, 130.42, 129.45, 129.17, 129.11, 129.03, 128.66, 128.54, 128.29, 127.54, 127.19, 83.80, 78.22, 75.61, 75.43, 58.13, 55.59, 50.26, 32.55, 32.23, 30.28, 29.96, 23.26, 14.29; HR-ESI-MS: m/z (ESI) calcd for $C_{36}H_{43}N_2$ $(M+H)^+$ 503.34208, found 503.34230

1f 1,3-dibenzyl-2,2-dimethyl-4,5-diphenylimidazolidine



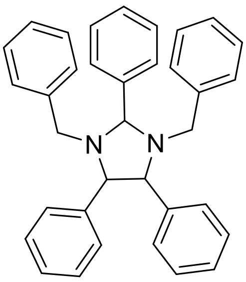
Prepared according to the general procedure A as described above in 25% yield. (monitored by TLC, petroleum ether:EtOAc=30:1, $R_f = 0.35$) It was purified by column chromatography (PE/EtOAc=150/1) to afford white solid. ^{1}H NMR (600 MHz, Acetone-*d$_6$*) δ 7.50-7.04 (m, 20H), 4.23 (q, 5.8 Hz, 0.44H), 4.19 (s, 1H), 4.03 (d, 8.4 Hz, 0.47H), 3.98 (d, 8.4 Hz, 0.45H), 3.94 (d, 13.4 Hz, 0.45H), 3.89 (d, 13.4 Hz, 1H), 3.80 (d, 4.5 Hz, 0.7H), 3.78 (d, 3.4 Hz, 0.5H), 3.62 (d, 9.3 Hz, 0.7H), 3.59 (d, 5.2 Hz, 0.3H), 1.40 (s, 6H); ^{13}C NMR (151 MHz,Acetonitrile-*d$_3$*) δ 140.94, 140.41, 139.84, 139.61, 139.53, 138.64, 129.05, 128.96, 128.41, 128.28, 128.21, 128.14, 128.09, 128.02, 127.99, 127.94, 127.71, 127.31, 126.93, 126.86, 126.66, 126.13, 81.24, 76.96, 75.75, 73.55, 70.80, 57.75, 55.24, 49.67, 22.27; HR-ESI-MS: m/z (ESI) calcd for $C_{31}H_{33}N_2$ $(M+H)^+$ 433.26383, found 433.26389

1g 1,3-dibenzyl-2-(tert-butyl)-4,5-diphenylimidazolidine



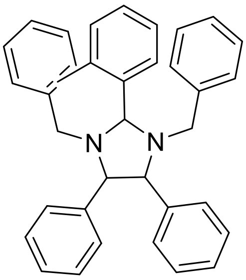
Prepared according to the general procedure A as described above in 48% yield. (monitored by TLC, petroleum ether:EtOAc=30:1, $R_f = 0.34$) It was purified by column chromatography (PE/EtOAc=100/1) to afford white solid. ^{1}H NMR (400 MHz, Acetone-*d$_6$*) δ 7.34-6.94 (m, 20H), 4.24 (s, 1H), 4.03 (d, 4.1 Hz, 0.5H), 3.82 (q, 13.7 Hz, 2H), 3.74 (2.6 Hz, 2H), 3.42 (d, 13.4 Hz, 1H), 3.40 (d, 13.3 Hz, 1H), 0.87 (s, 9H); ^{13}C NMR (100 MHz,Acetonitrile-*d$_3$*) δ 142.03, 140.77, 140.67, 140.31, 138.04, 130.66, 130.42, 129.45, 129.17, 129.11, 129.03, 128.67, 128.63, 128.54, 128.44, 128.29, 127.54, 127.19, 85.80, 75.58, 75.43, 56.27, 50.98, 32.55, 32.23, 23.26; HR-ESI-MS: m/z (ESI) calcd for $C_{33}H_{37}N_2$ $(M+H)^+$ 461.29513, found 461.29508

1h 1,3-dibenzyl-2,4,5-triphenylimidazolidine



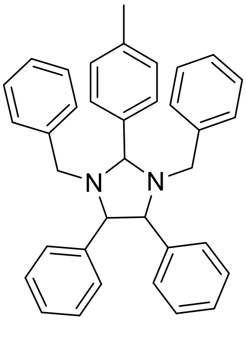
Prepared according to the general procedure B as described above in 98% yield. (monitored by TLC, petroleum ether:EtOAc=30:1, $R_f = 0.32$) It was purified by column chromatography (PE/EtOAc=150/1) to afford white solid. ^{1}H NMR (300 MHz, Acetone-*d₆*) δ 7.60-6.97 (m, 25H), 5.13 (s, 0.60H), 4.39 (d, 4.8 Hz, 0.48H), 4.33 (d, 11.3 Hz, 0.76H), 4.18 (d, 8.0 Hz, 0.75H), 3.90-3.71 (m, 2H), 3.62 (d, 14.1 Hz, 0.48H), 3.53 (d, 15.0 Hz, 0.73H), 3.18 (d, 14.9 Hz, 0.63H); ^{13}C NMR (150 MHz,Acetonitrile-d_3) δ 140.76, 140.71, 140.34, 140.06, 129.80, 129.20, 129.05, 129.04, 128.94, 128.42, 128.12, 127.77, 127.71, 127.34, 78.43,76.66, 76.25, 73.29, 58.15, 57.74; HR-ESI-MS: m/z (ESI) calcd for $C_{35}H_{33}N_2$ $(M+H)^+$ 481.26383, found 481.26358

1i 1,3-dibenzyl-4,5-diphenyl-2-(o-tolyl)imidazolidine



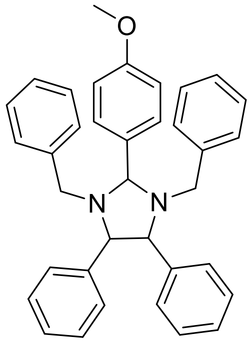
Prepared according to the general procedure B as described above in 65% yield. (monitored by TLC, petroleum ether:EtOAc=30:1, $R_f = 0.31$) It was purified by column chromatography (PE/EtOAc=150/1) to afford white oily solid. ^{1}H NMR (300 MHz, Acetone-d_6) δ 7.64-6.76 (m, 24H), 5.31 (dd, 6.4 Hz, 0.28H), 5.11 (dd, 9.5 Hz, 0.45H), 4.38-4.20 (m, 1H), 4.15-4.03 (m, 1H), 3.80-3.62 (m, 2H), 3.63-3.43 (m, 1H), 3.32-3.06 (m, 1H), 2.76 (s, 1H), 2.66 (s, 2H); ^{13}C NMR (100 MHz,Acetonitrile-d_3) δ 141.90, 140.82, 140.67, 140.40, 138.04, 130.66, 130.42, 129.46, 129.17, 129.11, 129.03, 128.67, 128.63, 128.54, 128.44, 128.29, 127.79, 127.54, 127.19, 83.72, 75.58, 75.43, 55.53,50.98, 23.26; HR-ESI-MS: m/z (ESI) calcd for $C_{36}H_{35}N_2$ $(M+H)^+$ 495.27948, found 495.27914

1j 1,3-dibenzyl-4,5-diphenyl-2-(p-tolyl)imidazolidine



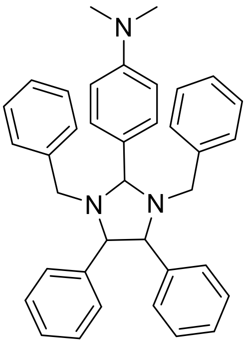
Prepared according to the general procedure B as described above in 74% yield. (monitored by TLC, petroleum ether:EtOAc=30:1, $R_f = 0.21$) It was purified by column chromatography (PE/EtOAc=50/1) to afford white oily solid. ^{1}H NMR (400 MHz, Acetonitrile-d_3) δ 7.50-6.77 (m, 24H), 5.01 (q, 7.3 Hz, 0.73H), 4.32-4.14 (m, 1.31H), 4.12-4.01 (m, 0.79H), 3.76-3.58 (m, 2H), 3.55-3.37 (m, 1H), 3.13-3.03 (m, 0.75H), 2.44 (s, 1.20H), 2.33 (s, 1.61H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 141.80, 140.66, 140.53, 140.46, 130.05, 129.81, 129.25, 129.16, 129.10, 128.90, 128.86, 128.71, 128.44, 128.07, 127.96, 127.69, 127.60, 127.46,78.15, 76.09, 74.71, 59.33, 23.58; HR-ESI-MS: m/z (ESI) calcd for $C_{36}H_{35}N_2$ (M+H)$^+$ 495.27948, found 495.27991

1k 1,3-dibenzyl-2-(4-methoxyphenyl)-4,5-diphenylimidazolidine



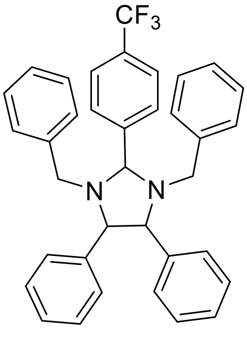
Prepared according to the general procedure B as described above in 97% yield. (monitored by TLC, petroleum ether:EtOAc=30:1, R_f = 0.20) It was purified by column chromatography (PE/EtOAc=50/1) to afford white oily solid. ^{1}H NMR (600 MHz, Acetone-d_6) δ 8.02-6.93 (m, 24H), 5.12 (d, 3.0 Hz, 0.32H), 5.08 (d, 2.8 Hz, 0.11H), 4.40-4.26 (m, 2H), 4.19-4.05 (m, 0.58H), 3.85-3.66 (m, 2H), 3.78 (s, 3H), 3.65-3.49 (m, 1.61H), 3.21-3.12 (m, 0.57H); ^{13}C NMR (151 MHz, Acetonitrile-d_3) δ 156.63， 141.04, 140.87, 140.77, 140.60, 140.52, 140.42, 140.35, 140.20, 140.07, 139.92, 139.80, 139.51, 139.44, 139.37, 139.32, 139.18, 139.06, 138.96, 138.87, 138.74, 138.69, 138.10, 137.09, 136.91, 136.71, 136.66, 136.52, 136.35, 136.25, 132.40, 132.10, 132.07, 131.64, 131.27, 131.21, 131.14, 131.06, 130.86, 130.41, 130.29, 130.19, 130.16, 130.13, 130.05, 129.99, 129.75, 129.61, 129.54, 129.16, 129.14, 128.65, 128.55, 128.36, 128.27, 128.23, 128.14, 127.79, 127.74, 127.66, 127.60, 127.57, 127.48, 127.41, 127.28, 127.22, 127.05, 126.99, 126.67, 126.59, 126.17, 121.85, 121.72, 121.63, 120.99, 120.94, 120.88, 120.78, 120.63, 119.61, 119.53, 119.32, 119.24, 86.16, 85.98, 85.84, 85.70, 85.62, 85.54, 83.18, 82.90, 82.65, 82.44, 75.11, 74.72, 74.54, 74.49, 74.24, 74.02, 73.95, 55.27, 55.18, 54.93, 54.80, 54.72, 54.65, 54.58, 54.52, 54.25, 54.05, 53.91, 53.76, 53.65, 53.44, 53.01, 50.35, 50.31, 50.10, 50.07**;** HR-ESI-MS: m/z (ESI) calcd for $C_{36}H_{35}N_2O$ $(M+H)^+$ 511.27439, found 511.27407

1l 4-(1,3-dibenzyl-4,5-diphenylimidazolidin-2-yl)-N,N-dimethylaniline



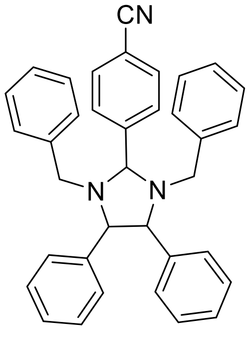
Prepared according to the general procedure B as described above in 58% yield. (monitored by TLC, petroleum ether:EtOAc=30:1, $R_f = 0.22$) It was purified by column chromatography (PE/EtOAc=100/1) to afford white solid. ^{1}H NMR (400 MHz, Acetonitrile-*d₃*) δ7.36-6.75 (m, 24H), 4.25-3.90 (m, 2H), 3.75-3.58 (m, 3H), 3.35-3.26 (m, 2H), 2.67 (s, 6H); ^{13}C NMR (100 MHz, Acetone-*d₆*) δ 150.92, 141.37, 140.26, 140.22, 139.88, 139.58, 130.13, 130.00, 129.92, 129.45, 129.28, 128.90, 128.68, 128.58, 128.53, 128.50, 128.47, 128.44, 128.09, 128.01, 127.98, 127.97, 127.90, 127.87, 127.85, 127.75, 127.61, 127.32, 127.25, 127.18, 127.03, 126.82, 126.49, 111.27, 86.07, 83.19, 77.94, 75.76, 77.11, 75.09, 72.81, 70.09, 57.65, 57.25, 54.94, 53.81, 50.92, 50.62, 40.44; HR-ESI-MS: m/z (ESI) calcd for $C_{37}H_{38}N_3$ $(M+H)^+$ 524.30602, found 524.30588

1m 1,3-dibenzyl-4,5-diphenyl-2-(4-(trifluoromethyl)phenyl)imidazolidine



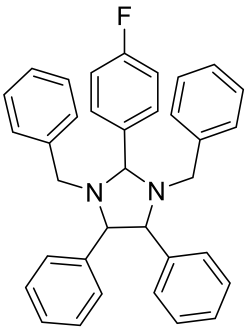
Prepared according to the general procedure B as described above in 70% yield. (monitored by TLC, petroleum ether:EtOAc=30:1, $R_f = 0.20$) It was purified by column chromatography (PE/EtOAc=50/1) to afford white solid. ^{1}H NMR (300 MHz, Acetonitrile-*d*$_3$) δ 7.96-6.83 (m, 24H), 5.12-5.03 (m, 0.54H), 4.40-3.97 (m, 2H), 3.75-3.66 (m, 2H), 3.58-3.39 (m, 2H), 3.11(t, J=3.5Hz, 0.54H); ^{13}C NMR (151 MHz, Acetonitrile-*d*$_3$) δ 145.78, 145.13, 144.76, 144.36, 144.24, 141.03, 140.83, 140.76, 140.58, 140.52, 140.42, 140.19, 139.91, 139.80, 139.49, 139.30, 139.18, 139.07, 138.99, 137.09, 137.05, 136.98, 136.82, 136.66, 136.06, 134.44, 130.15, 130.13, 129.81, 129.75, 129.61, 129.53, 129.45, 129.16, 129.11, 129.05, 128.85, 128.77, 128.57, 128.54, 128.34, 128.30, 128.26, 128.23, 128.14, 127.93, 127.82, 127.78, 127.74, 127.67, 127.65, 127.55, 127.45, 127.40, 127.24, 126.99, 126.77, 126.72, 126.65, 126.61, 126.59, 126.49, 126.47, 126.39, 126.31, 126.17, 125.06, 124.95, 124.93, 123.64, 123.49, 123.47, 85.98, 85.70, 83.59, 83.46, 83.01, 82.90, 74.72, 74.54, 74.46, 74.28, 74.14, 69.59, 69.46, 69.35, 55.20, 55.01, 54.64, 54.43, 53.44, 52.81, 50.61, 50.51, 50.10, 50.02; HR-ESI-MS: m/z (ESI) calcd for $C_{36}H_{32}F_3N_2$ $(M+H)^+$ 549.25121, found 549.25102

1n 4-(1,3-dibenzyl-4,5-diphenylimidazolidin-2-yl)benzonitrile



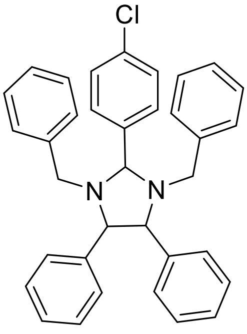
Prepared according to the general procedure B as described above in 70% yield. (monitored by TLC, petroleum ether:EtOAc=15:1, R_f = 0.20) It was purified by column chromatography (PE/EtOAc=30/1) to afford white solid. ^{1}H NMR (400 MHz, Acetone-*d*$_6$) δ 7.96-6.85 (m, 24H), 5.07 (s, 0.5H), 4.36 (s, 0.5H), 4.29-4.23 (m, 1H), 4.12 (d, 8.5 Hz, 0.5H), 3.79-3.68 (m, 1H), 3.66 (s, 0.5H), 3.57 (s, 0.5H), 3.55-3.44 (m, 1H), 3.10 (d, 14.4 Hz, 0.5H); ^{13}C NMR (100 MHz, Acetone-*d*$_6$) δ141.35, 141.14, 140.40, 140.31, 140.19, 139.76, 137.24, 136.92, 134.65, 130.48, 130.12, 129.98, 129.90, 129.73, 129.43, 128.89, 128.85, 128.57, 128.49, 128.46, 128.08, 128.07, 128.00, 127.97, 127.95, 127.86, 127.84, 127.74, 127.30, 127.01, 126.90, 126.63, 126.49, 108.78, 108.21, 86.07, 83.18, 75.10, 75.08, 70.03, 54.95, 53.82, 50.64; HR-ESI-MS: m/z (ESI) calcd for $C_{36}H_{32}N_3$ $(M+H)^+$ 506.25907, found 506.25922

1o 1,3-dibenzyl-2-(4-fluorophenyl)-4,5-diphenylimidazolidine



Prepared according to the general procedure B as described above in 51% yield. (monitored by TLC, petroleum ether:EtOAc=30:1, R_f = 0.18) It was purified by column chromatography (PE/EtOAc=50/1) to afford white solid. ^{1}H NMR (300 MHz, Acetonitrile-d_3) δ 7.83-6.75 (m, 24H), 4.92 (d, 6.1 Hz, 0.67H), 4.42 (s, 0.14H), 4.21-4.08 (m, 1.43H), 4.01-3.86 (m, 0.73H), 3.66-3.46 (m, 2H), 3.46-3.28 (m, 1.26H), 2.97 (dd, 4.9 Hz, 0.67H); ^{13}C NMR (151 MHz, Acetonitrile-d_3) δ 162.95, 161.33, 141.03, 140.76, 140.19, 140.14, 139.90, 139.79, 139.49, 137.17, 137.08, 136.98, 136.79, 136.64, 131.99, 131.93, 131.52, 131.48, 130.16, 130.00, 129.76, 129.71, 129.62, 129.53, 129.16, 129.11, 128.55, 128.51, 128.47, 128.27, 128.24, 128.15, 127.83, 127.78, 127.75, 127.65, 127.55, 127.44, 127.40, 127.05, 126.99, 126.65, 126.60, 126.56, 126.38, 126.31, 126.25, 126.18, 115.06, 114.92, 114.88, 114.83, 114.76, 114.74, 114.69, 114.62, 113.95, 113.90, 113.81, 113.76, 85.67, 85.46, 82.87, 82.36, 74.99, 74.71, 74.53, 74.41, 71.11, 69.75, 69.57, 55.06, 54.95, 54.80, 53.68, 53.40, 50.23, 50.20, 50.14, 50.11; HR-ESI-MS: m/z (ESI) calcd for $C_{35}H_{32}FN_2$ $(M+H)^+$ 499.25440, found 499.25433

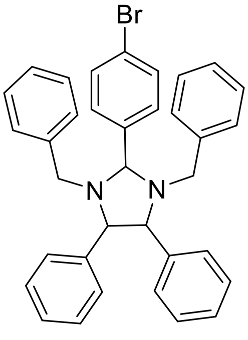
1p 1,3-dibenzyl-2-(4-chlorophenyl)-4,5-diphenylimidazolidine



Prepared according to the general procedure B as described above in 53% yield.

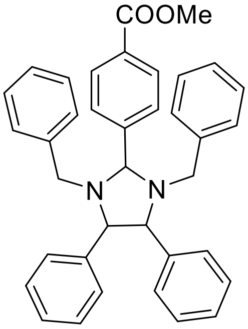
(monitored by TLC, petroleum ether:EtOAc=30:1, $R_f = 0.23$) It was purified by column chromatography (PE/EtOAc=100/1) to afford white solid. ^{1}H NMR (400 MHz, Acetone-*d₆*) δ 7.48-6.86 (m, 24H), 5.08 (s, 1H), 4.32-4.22 (m, 1H), 4.14-4.02 (m, 1H), 3.81-3.61 (m, 2H), 3.61-3.44 (m, 2H); ^{13}C NMR (100 MHz, Acetone-*d₆*) δ 140.97, 140.01, 139.38, 139.26, 139.11, 136.85, 136.66, 136.54, 131.30, 131.27, 130.56, 130.10, 130.08, 129.77, 129.74, 129.72, 129.60, 129.52, 129.06, 129.03, 128.50, 128.31, 128.29, 128.24, 127.74, 127.70, 127.67, 127.64, 127.62, 127.59, 127.56, 127.50, 127.48, 127.46, 127.43, 27.39, 127.35, 126.92, 126.77, 126.67, 126.63, 126.52, 126.29, 126.24, 126.10, 83.10, 82.79, 74.71, 74.69, 74.19, 74.16, 74.11, 69.64, 54.55, 54.39, 53.43, 50.25, 50.19; HR-ESI-MS: m/z (ESI) calcd for $C_{35}H_{32}ClN_2$ $(M+H)^+$ 515.22485, found 515.22478

1q 1,3-dibenzyl-2-(4-bromophenyl)-4,5-diphenylimidazolidine



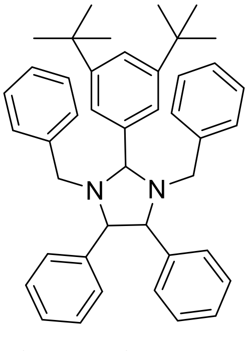
Prepared according to the general procedure B as described above in 62% yield. (monitored by TLC, petroleum ether:EtOAc=10:1, $R_f = 0.21$) It was purified by column chromatography (PE/EtOAc=20/1) to afford white solid. ^{1}H NMR (600 MHz, Acetonitrile-*d₃*) δ 7.63-6.83 (m, 24H), 5.05 (d, 3.0Hz, 0.35H), 5.00 (d, 3.0 Hz, 0.12H), 4.32-4.18 (m, 2H), 4.12-3.97 (m, 0.62H), 3.81-3.58 (m, 2H), 3.57-3.41 (m, 1.6H), 3.13-3.03 (m, 0.57H); ^{13}C NMR (151 MHz, Acetonitrile-*d₃*) δ 141.02, 140.88, 140.75, 140.58, 140.27, 140.23, 140.18, 140.09, 140.06, 139.92, 139.80, 139.52, 139.44, 139.36, 139.32, 139.18, 139.05, 138.96, 137.08, 136.91, 136.71, 136.67, 136.51, 136.35, 136.25, 135.52, 134.43, 132.39, 132.10, 132.06, 131.63, 131.25, 131.20, 131.14, 131.06, 130.41, 130.29, 130.19, 130.15, 130.13, 130.05, 129.98, 129.76, 129.72, 129.61, 129.53, 129.17, 129.14, 128.55, 128.26, 128.22, 128.14, 127.78, 127.73, 127.65, 127.59, 127.56, 127.47, 127.40, 127.05, 126.99, 126.67, 126.59, 126.35, 126.18, 122.21, 121.85, 121.75, 121.62, 121.01, 121.88, 121.76, 121.62, 119.62, 119.53, 119.34, 119.24, 85.97, 85.84, 85.70, 85.62, 85.55, 83.19, 82.91, 82.65, 82.45, 75.10,74.72, 74.54, 74.48, 74.23, 74.03, 73.95, 73.81, 69.83, 69.60, 69.30, 69.15, 69.06, 68.94, 68.89, 54.92, 54.81, 54.64, 54.26, 54.06, 53.90, 53.76, 53.64, 53.43, 53.01, 50.32, 50.08; HR-ESI-MS: m/z (ESI) calcd for $C_{35}H_{32}BrN_2$ $(M+H)^+$ 559.17434, found 559.16984

1r methyl 4-(1,3-dibenzyl-4,5-diphenylimidazolidin-2-yl)benzoate



Prepared according to the general procedure B as described above in 32% yield. (monitored by TLC, petroleum ether:EtOAc=10:1, $R_f = 0.18$) It was purified by column chromatography (PE/EtOAc=20/1) to afford white solid. ^1H NMR (400 MHz, Acetone-*d6*) δ 7.96-6.85 (m, 24H), 5.08 (s, 0.56H), 4.37 (s, 0.40H), 4.30-4.23 (m, 1H), 4.12 (d, 8.3 Hz, 0.76H), 3.93 (s, 3H), 3.79-3.45 (m, 3H), 3.10 (d, 14.9 Hz, 0.54H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 166.26, 145.53, 141.80, 140.66, 140.53, 140.46, 130.05, 129.81, 129.25, 129.16, 129.10, 128.90, 128.86, 128.71, 128.44, 128.07, 127.96, 127.69, 127.60, 127.46,86.69, 78.15, 76.09, 74.71, 59.33, 50.44; HR-ESI-MS: m/z (ESI) calcd for $C_{37}H_{35}N_2O_2$ $(M+H)^+$ 539.26930, found 539.26922

1s 1,3-dibenzyl-2-(3,5-di-tert-butylphenyl)-4,5-diphenylimidazolidine

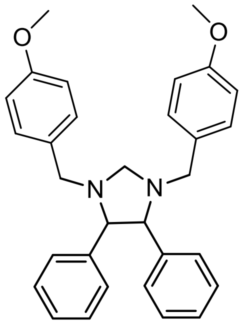


Prepared according to the general procedure B as described above in 48% yield.

(monitored by TLC, petroleum ether:EtOAc=10:1, $R_f = 0.24$) It was purified by column chromatography (PE/EtOAc=20/1) to afford white solid. ^{1}H NMR (400 MHz, Acetonitrile-*d₃*) δ 7.49-6.85(m, 23H), 5.08(s, 0.5H), 5.04(s, 0.5H), 4.40-4.17(m, 1H), 4.16-3.99(m, 1H), 3.98-3.76(m, 1H), 3.76-3.67(m, 1H), 3.67-3.43(m, 2H), 1.29(s,9H), 1.25(s, 9H) ^{13}C NMR (75 MHz, Acetone-d_6) δ 152.15, 141.22, 140.22, 138.00, 137.19, 137.11, 130.11, 129.91, 129.70, 129.42, 128.99, 128.88, 128.84, 128.57, 128.50, 128.47, 128.42, 128.10, 128.07, 128.00, 127.97, 127.73, 127.33, 127.31, 127.02, 126.78, 126.63, 124.27, 124.06, 84.41, 83.08, 75.04, 54.85, 50.71, 50.60, 34.59, 31.29

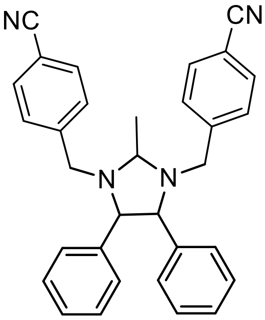
HR-ESI-MS: m/z (ESI) calcd for $C_{43}H_{49}N_2$ $(M+H)^+$ 593.38903, found 593.38912

1t 1,3-bis(4-methoxybenzyl)-4,5-diphenylimidazolidine



Prepared according to the general procedure B as described above in 78% yield. (monitored by TLC, petroleum ether:EtOAc=50:1, $R_f = 0.24$) It was purified by column chromatography (PE/EtOAc=50/1) to afford white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99-6.82(m, 18H), 5.05(t, 0.31H), 4.99(s, 6H), 4.78-4.82(d, 0.34H), 4.62-4.67 (d, 0.53H), 4.35-4.23 (m, 0.58H), 4.21-4.07 (m, 0.64H), 4.03-3.95 (dd, 0.23H), 3.73-3.60 (m, 1.16H), 3.53-3.41 (m, 0.85H), 3.16-3.10 (m, 0.30H) HR-ESI-MS: m/z (ESI) calcd for $C_{31}H_{33}N_2O_2$ $(M+H)^+$ 464.24638, found 464.24632.

1u4,4'-(2-methyl-4,5-diphenylimidazolidine-1,3-diyl)bis(methylene)dibenzonitrile



Prepared according to the general procedure B as described above in 62% yield.

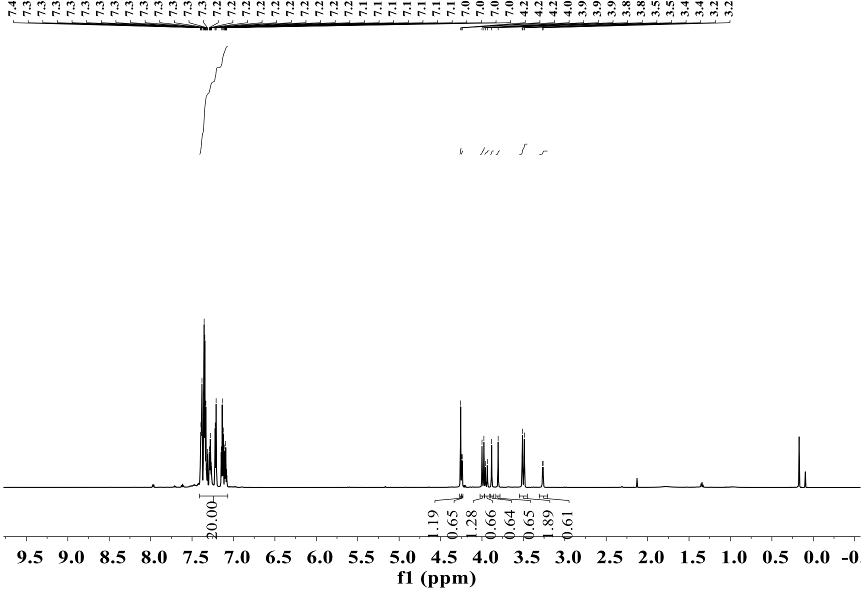
(monitored by TLC, petroleum ether:EtOAc=30:1, $R_f = 0.21$) It was purified by column chromatography (PE/EtOAc=30/1) to afford white solid. ^{1}H NMR (400 MHz, CD_3OD) δ 7.91-6.84(m, 18H), 4.99(s, 0.28H), 4.70-4.62(t, 0.28H), 4.27-4.00 (m, 1.36H), 3.73-3.60 (m, 1.01H), 3.51-3.41 (m, 0.72H), 3.30 (q, 2.26H), 3.05-3.01 (d, 0.39H), 0.89-0.86 (d, 3H) HR-ESI-MS: m/z (ESI) calcd for $C_{32}H_{29}N_4$ $(M+H)^+$ 468.23140, found 468.23136.

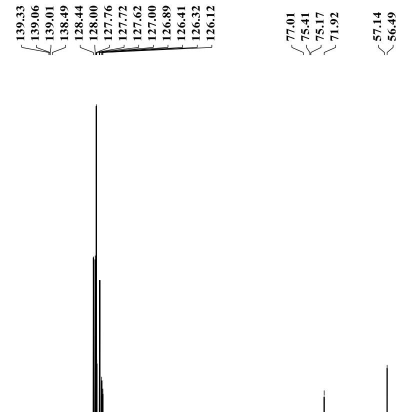
3c

Prepared according to the general procedure B as described above in 92% yield. (monitored by TLC, petroleum ether:EtOAc=50:1, $R_f = 0.24$) It was purified by column chromatography (PE/EtOAc=50/1) to afford white solid. ^{1}H NMR (400 MHz, Acetone-*d$_6$*) δ 768-7.34(m, 10H), 5.09(d, 0.47H), 4.71(s, 0.28H), 4.48 (d, 0.68H), 4.08 (d, 0.66H), 3.77(s, 1.58H), 3.71-3.60 (m, 2.45H), 3.51 (s, 1.38H), 2.09-2.05 (m, 5H), 1.11 (d, 3H); ^{13}C-NMR (100 MHz, acetone-*d$_6$*) δ 169.51, 165.39, 144.44, 140.82, 130.50, 130.08, 129.10, 128.93, 128.85, 128.68, 128.45, 128.36, 128.12, 128.04, 127.21, 83.55, 76.77, 68.80, 51.53, 51.18, 47.46, 46.81, 22.43; HR-ESI-MS: m/z (ESI) calcd for $C_{22}H_{27}N_2O_4$ $(M+H)^+$ 382.18926, found 382.18929.

IX. ^{1}H NMR and ^{13}C NMR spectra of all cycloaddition products

1a





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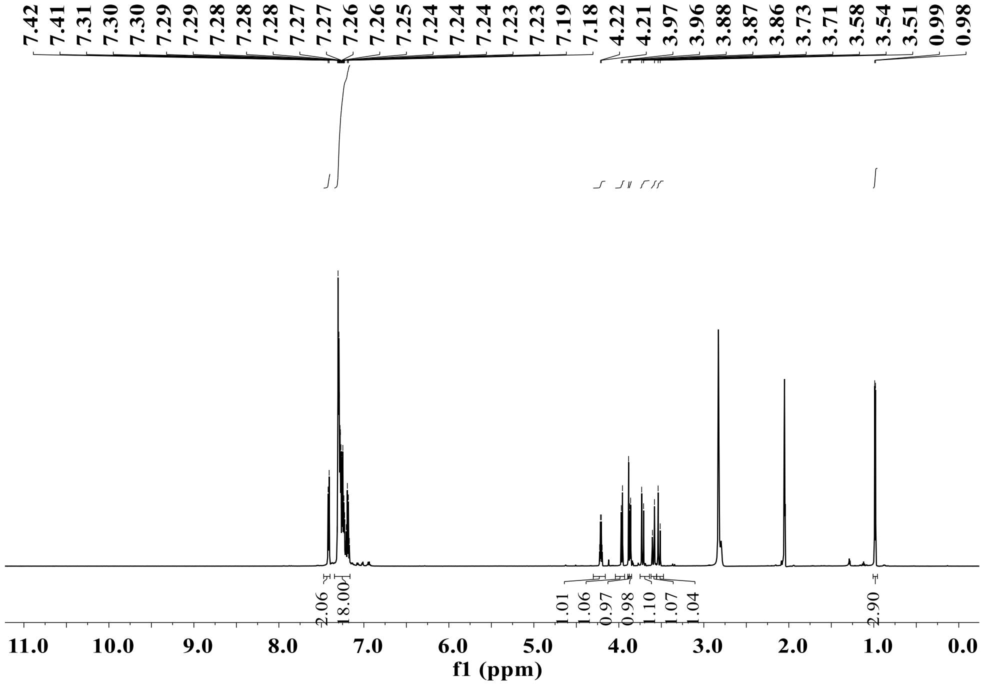
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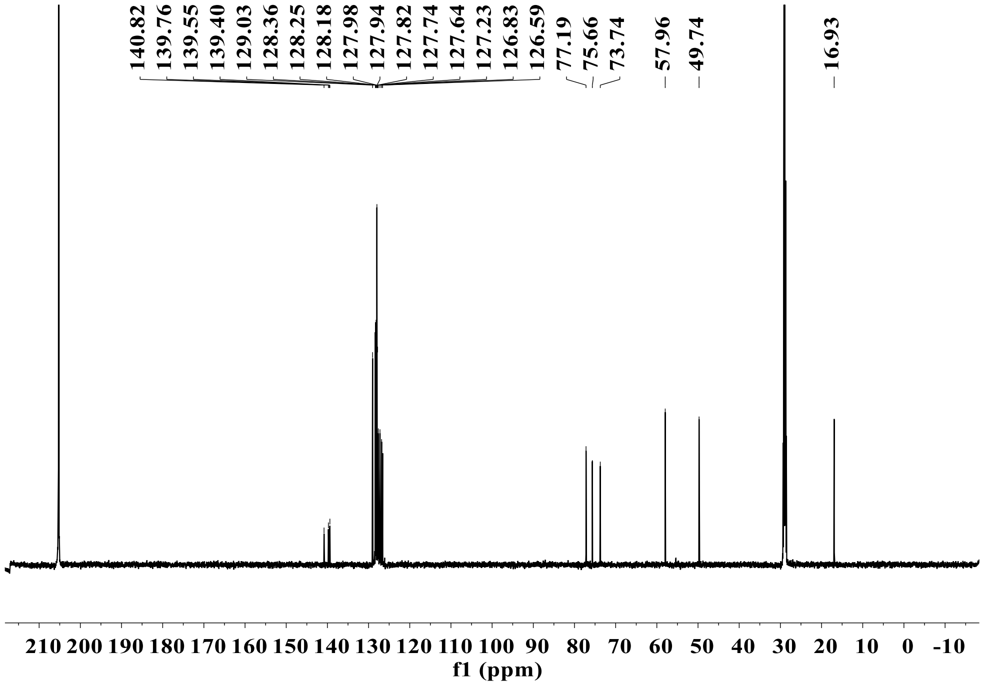
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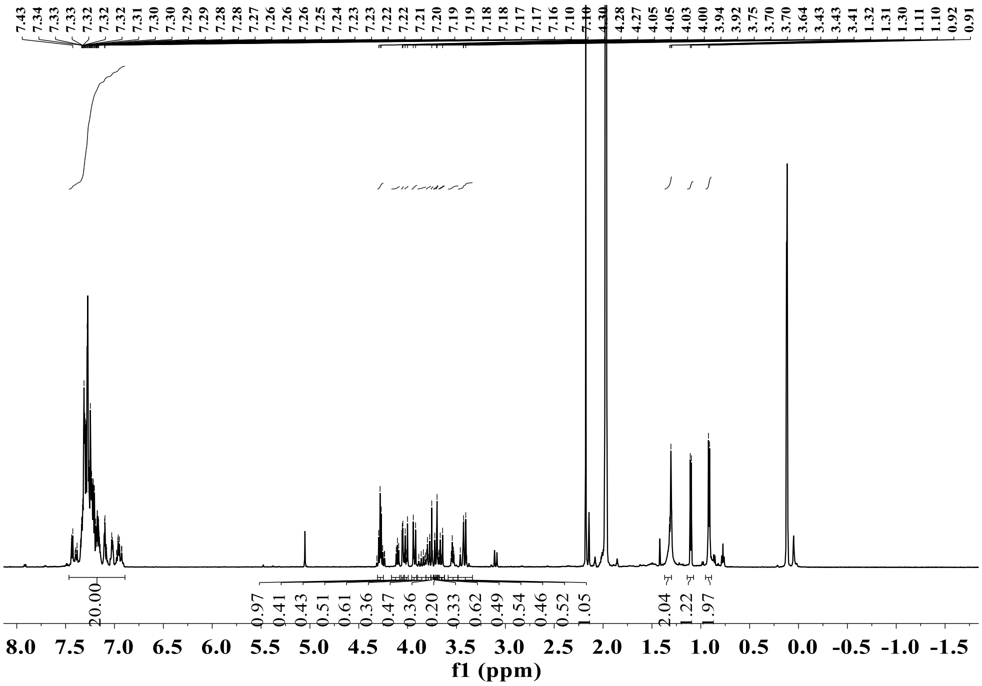
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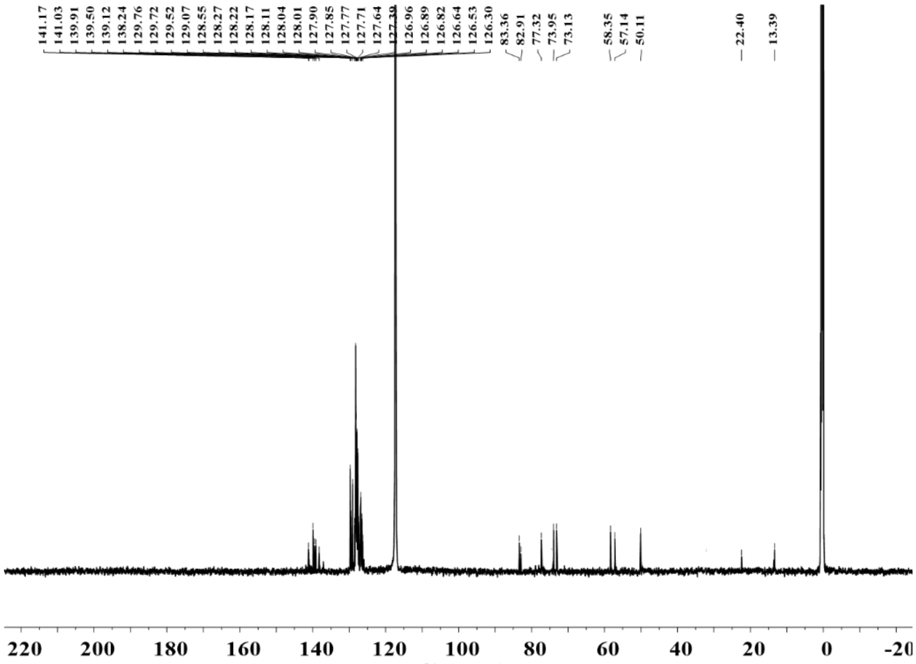
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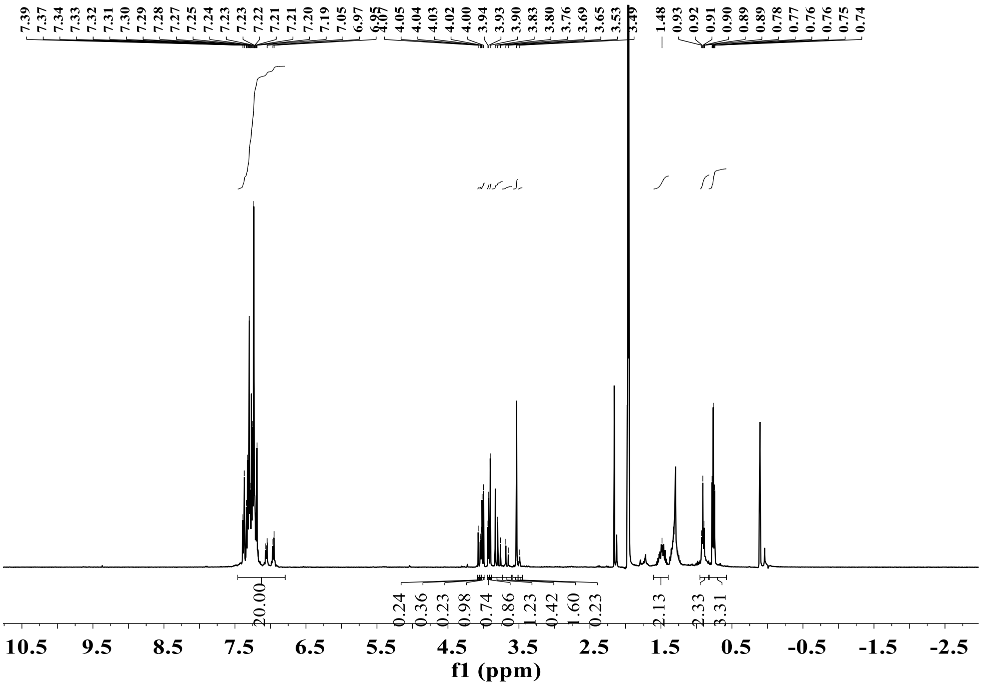
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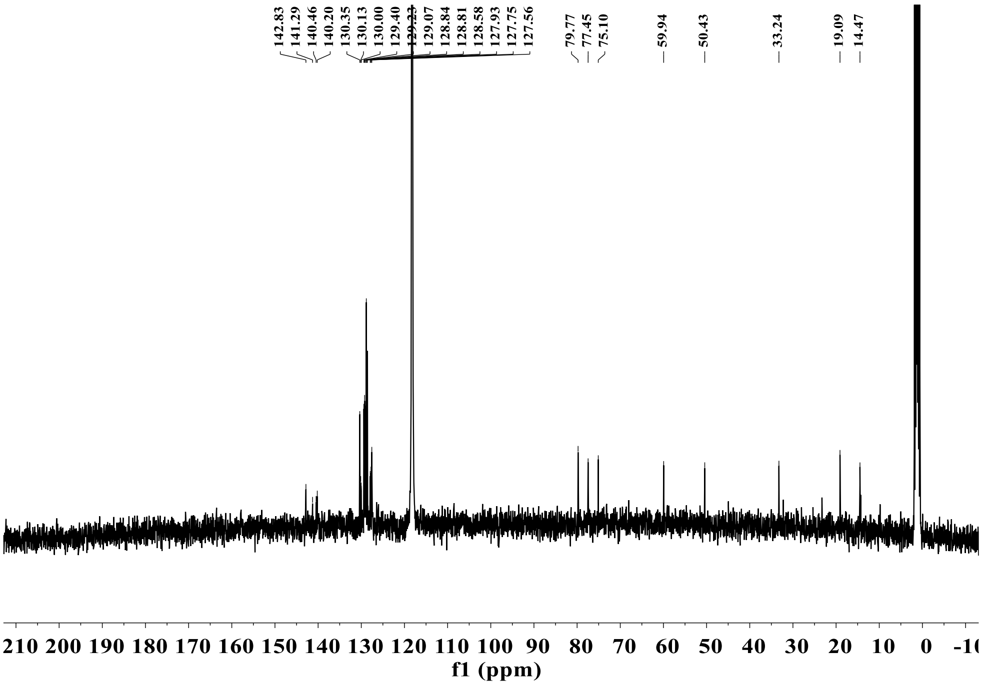
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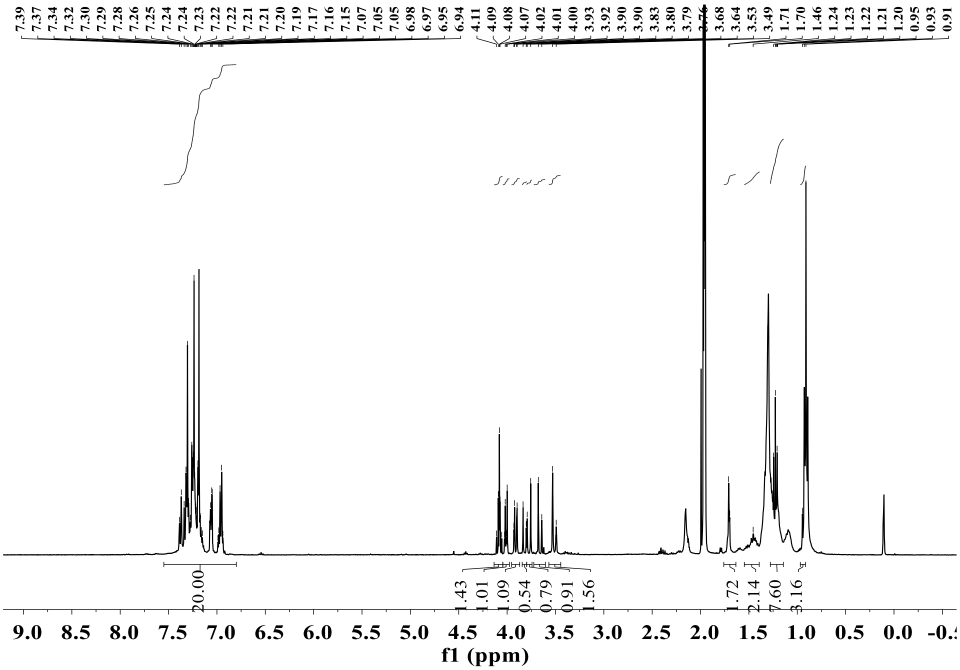
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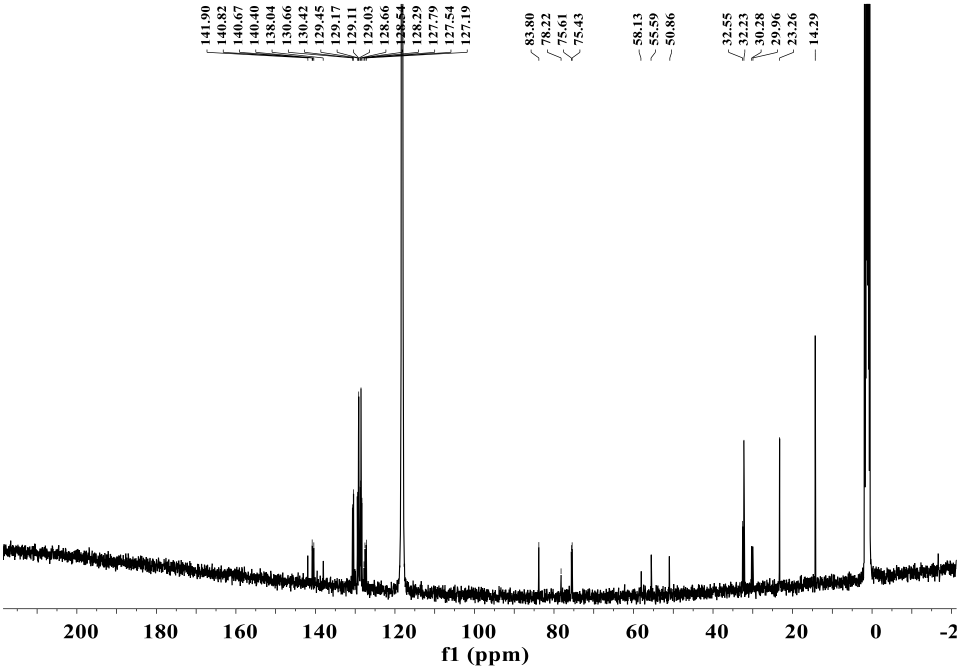
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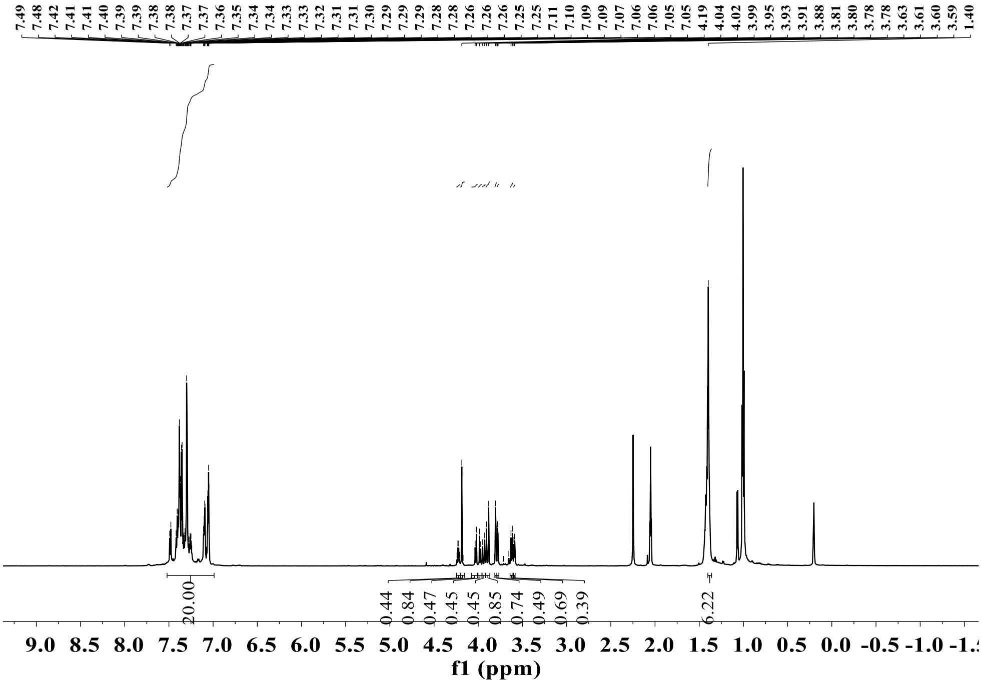
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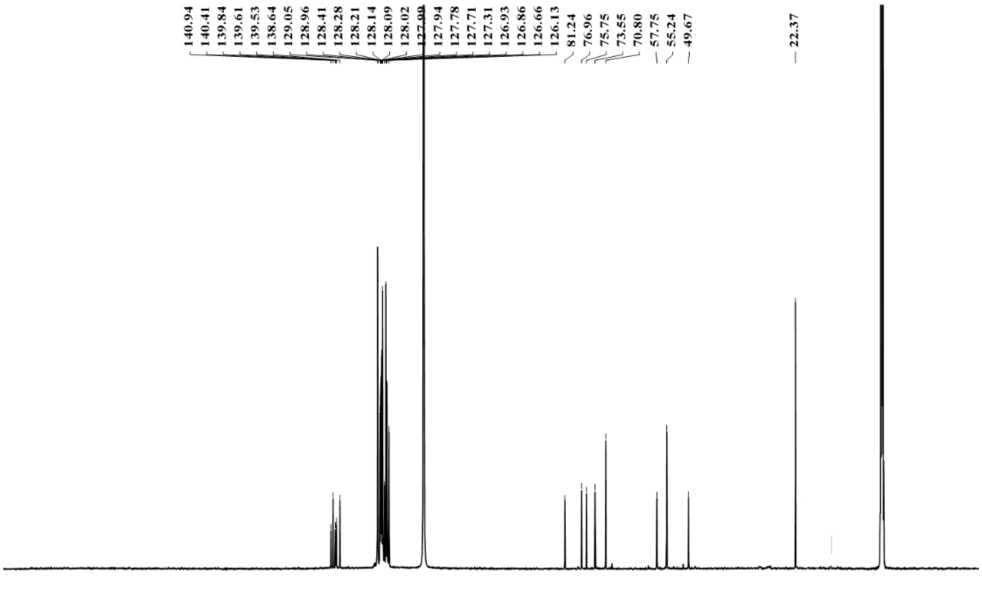
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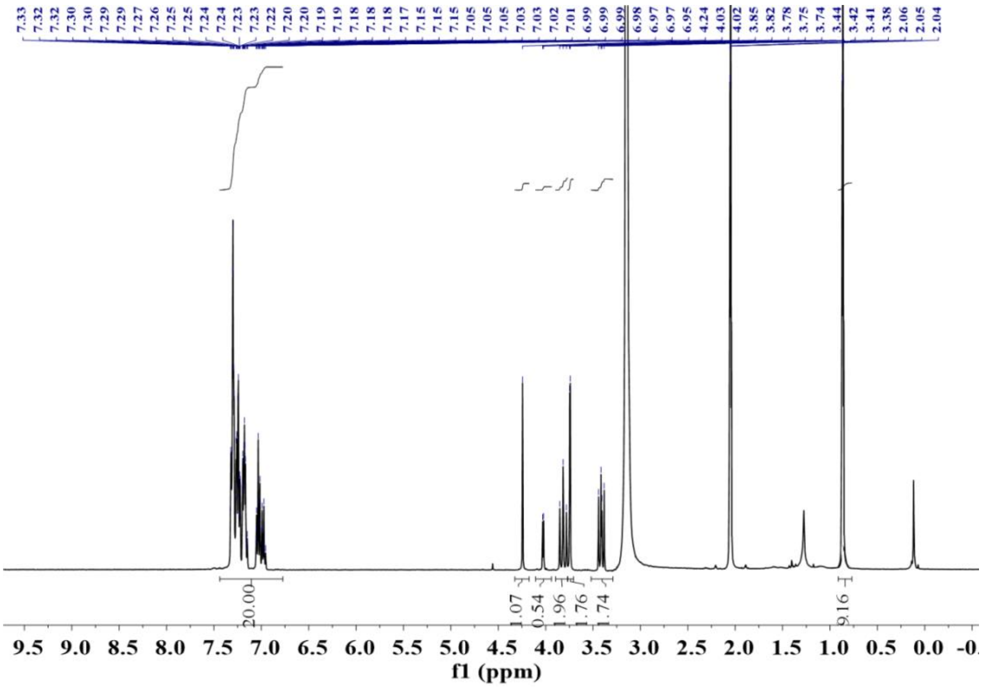
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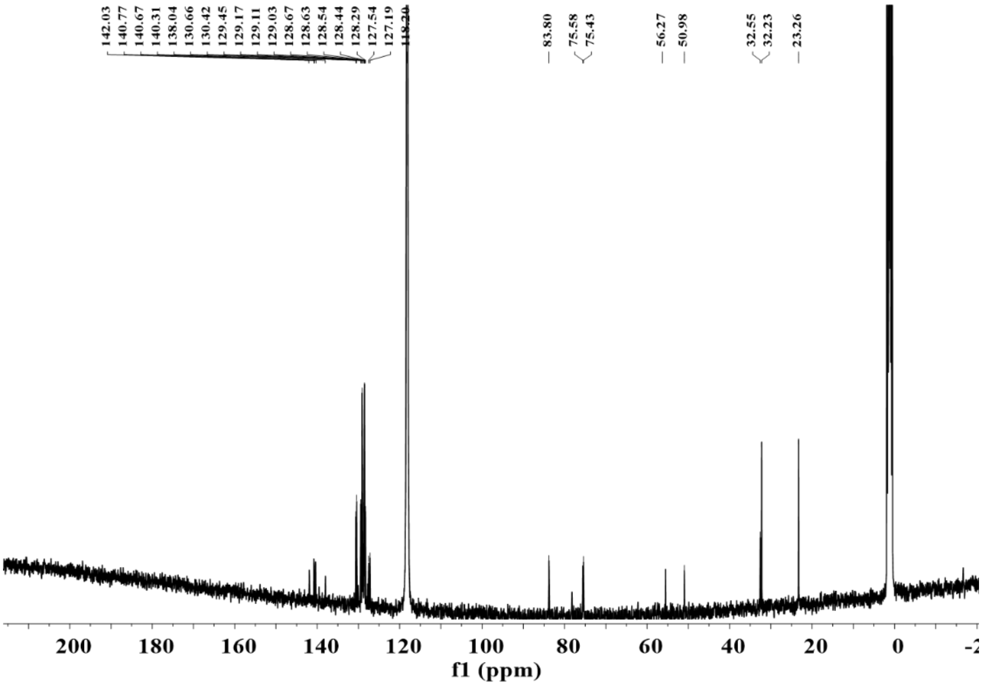
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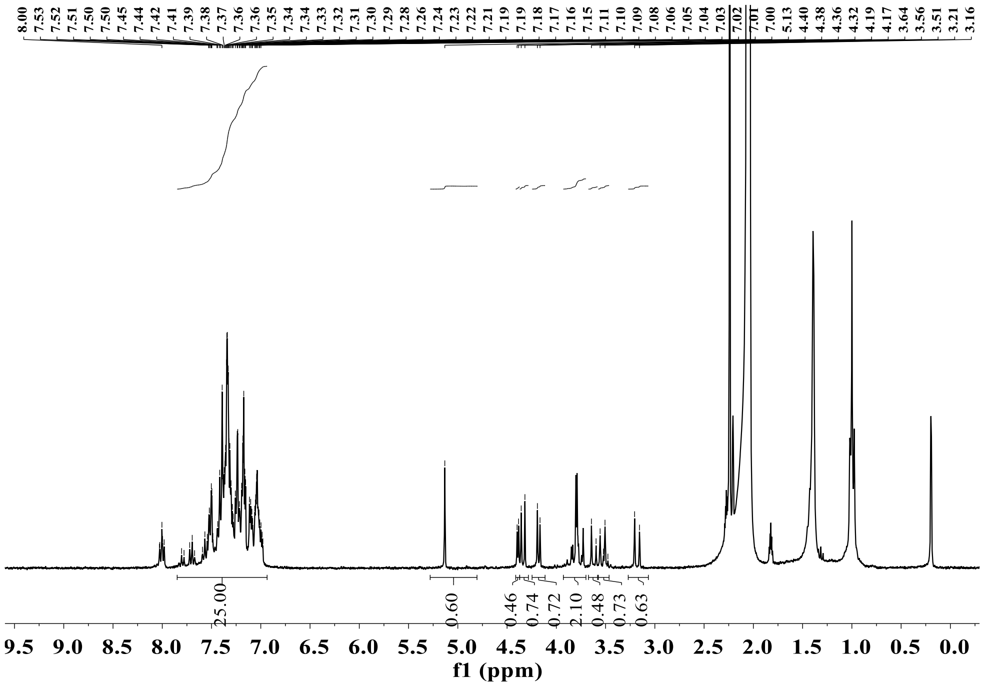
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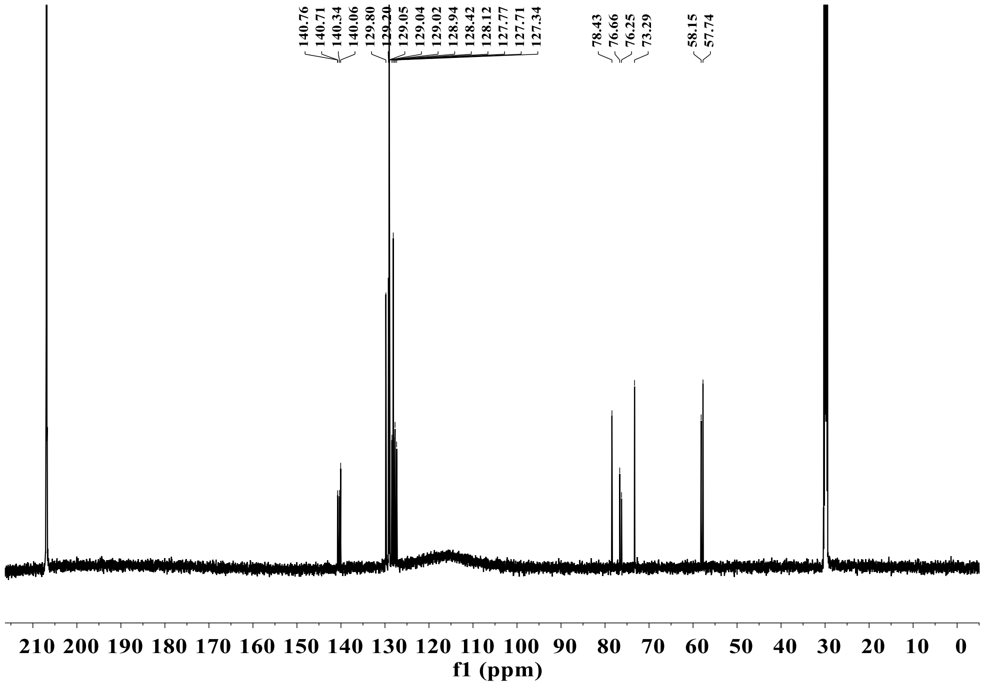
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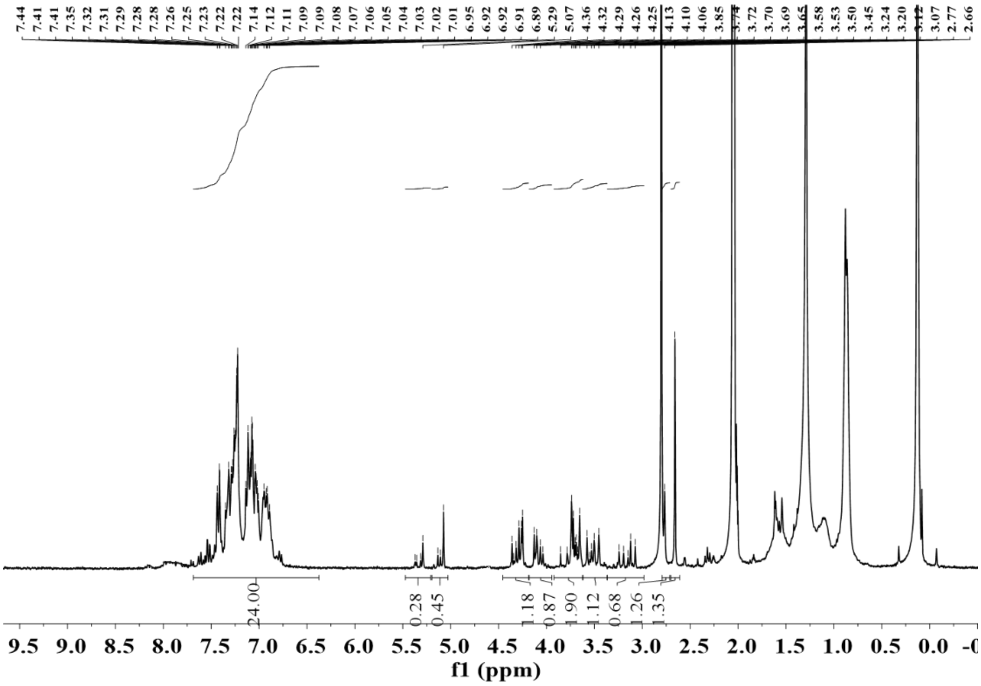
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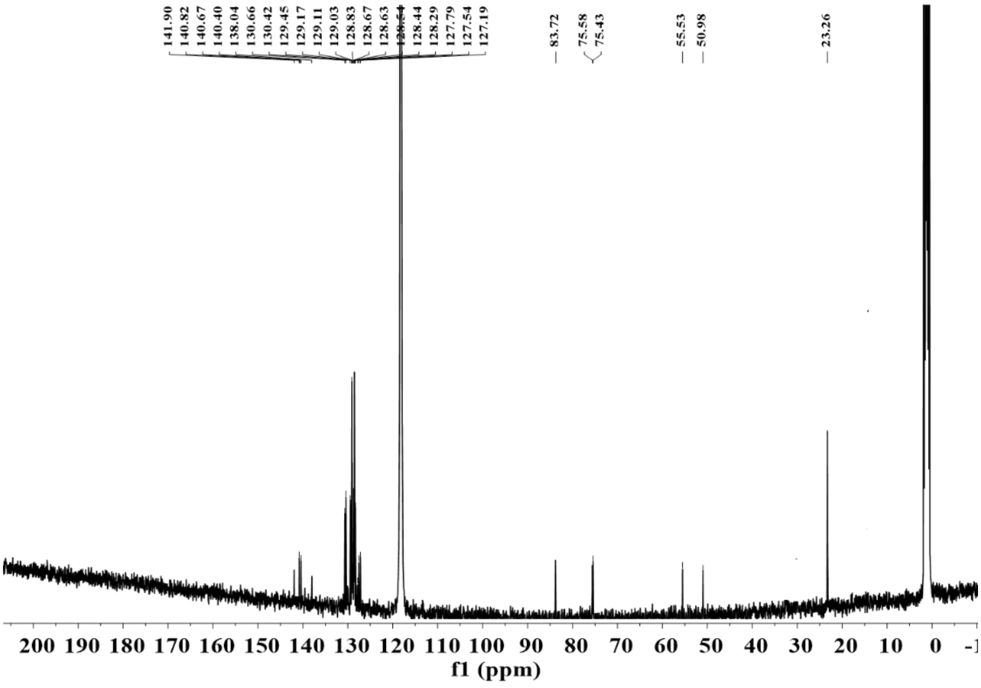
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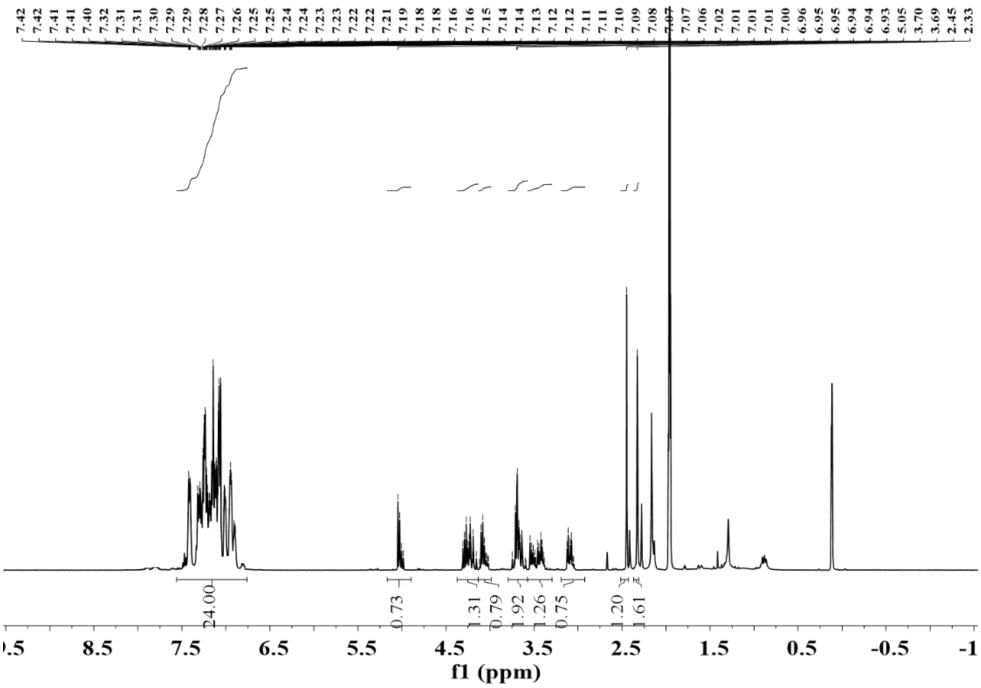
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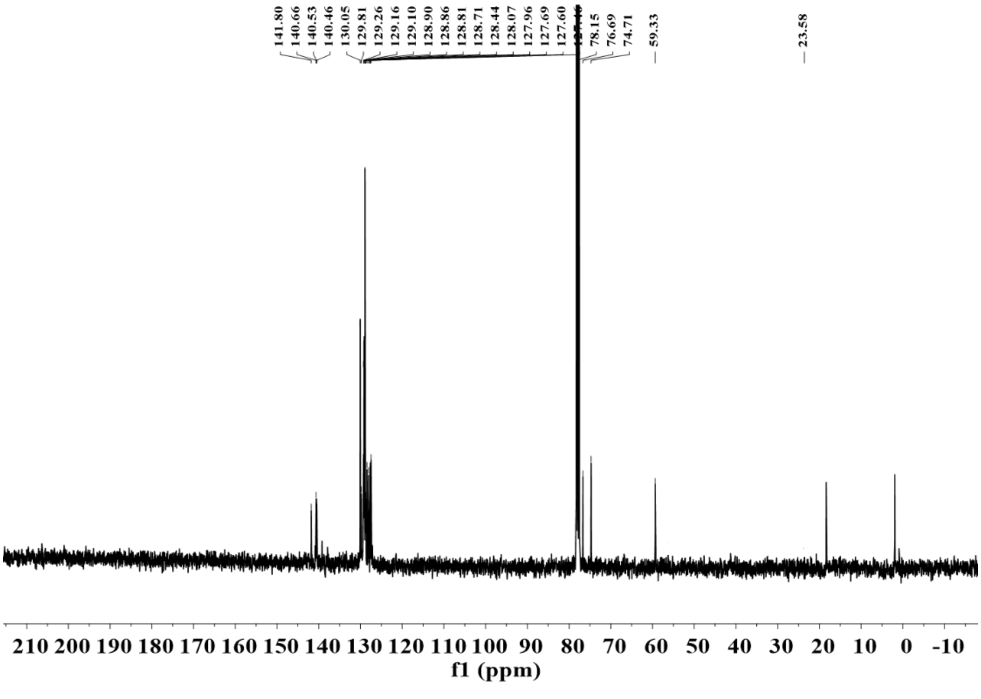
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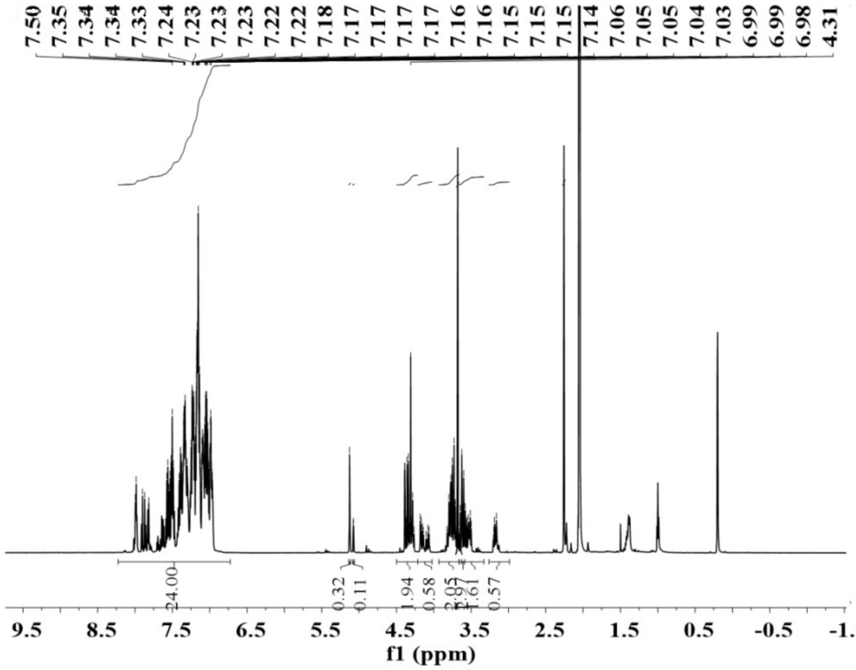
f1 (ppm)

1k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7.16
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7.15
7.15
7.14
7.06
7.05
7.05
7.04
7.03
6.99
6.99
6.98
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24.00
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0.11
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0.58
2.05
2.97
1.61
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8.5
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4.5
3.5
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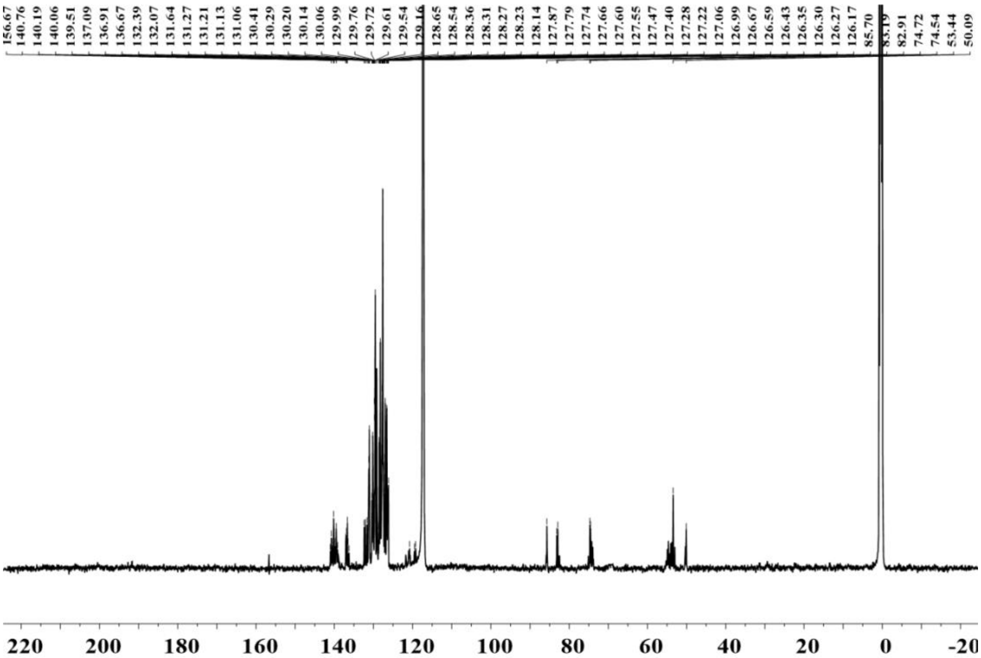
-1.5
f1 (ppm)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126.27
126.17
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82.91
74.72
74.54
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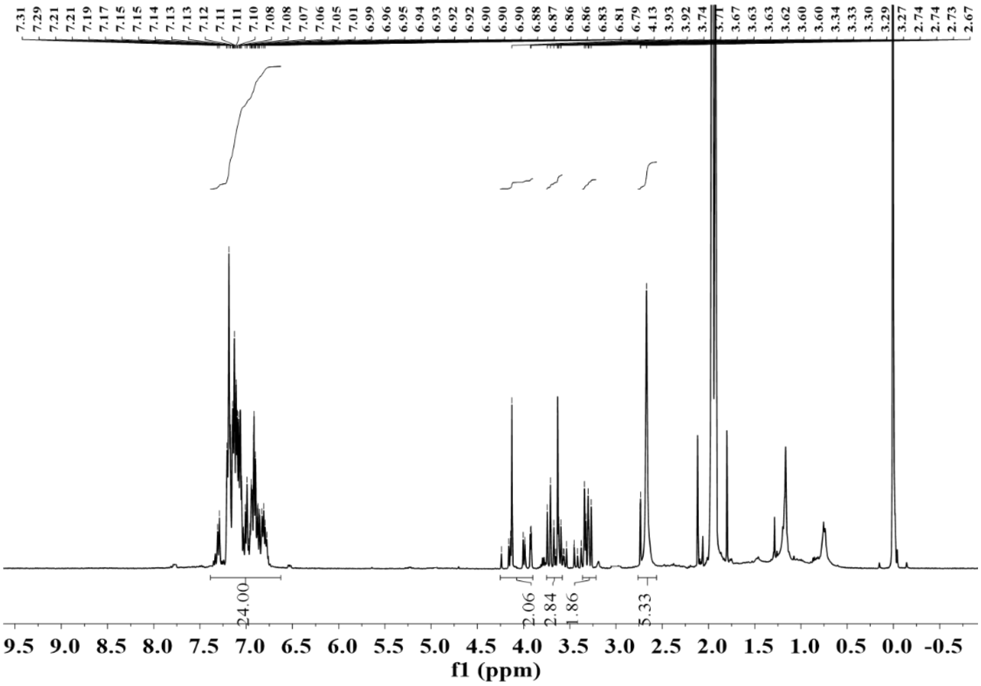
f1 (ppm)

11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7.05
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6.95
6.94
6.93
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3.62
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3.29
3.27
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2.74
2.73
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2.06
2.84
1.86
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8.0
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7.0
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6.0
5.5
5.0
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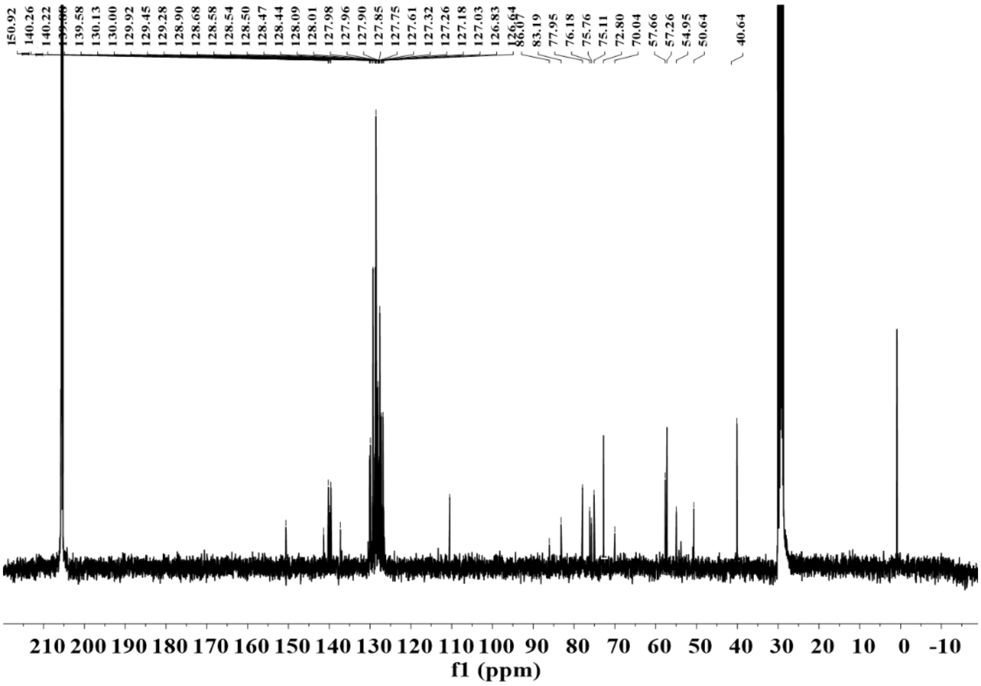
f1 (ppm)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128.54
128.50
128.47
128.44
128.09
128.01
127.98
127.96
127.90
127.85
127.75
127.61
127.32
127.26
127.18
127.03
126.83
126.64
86.07
83.19
77.95
76.18
75.76
75.11
72.80
70.04
57.66
57.26
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40.64
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200
190
180
170
160
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20
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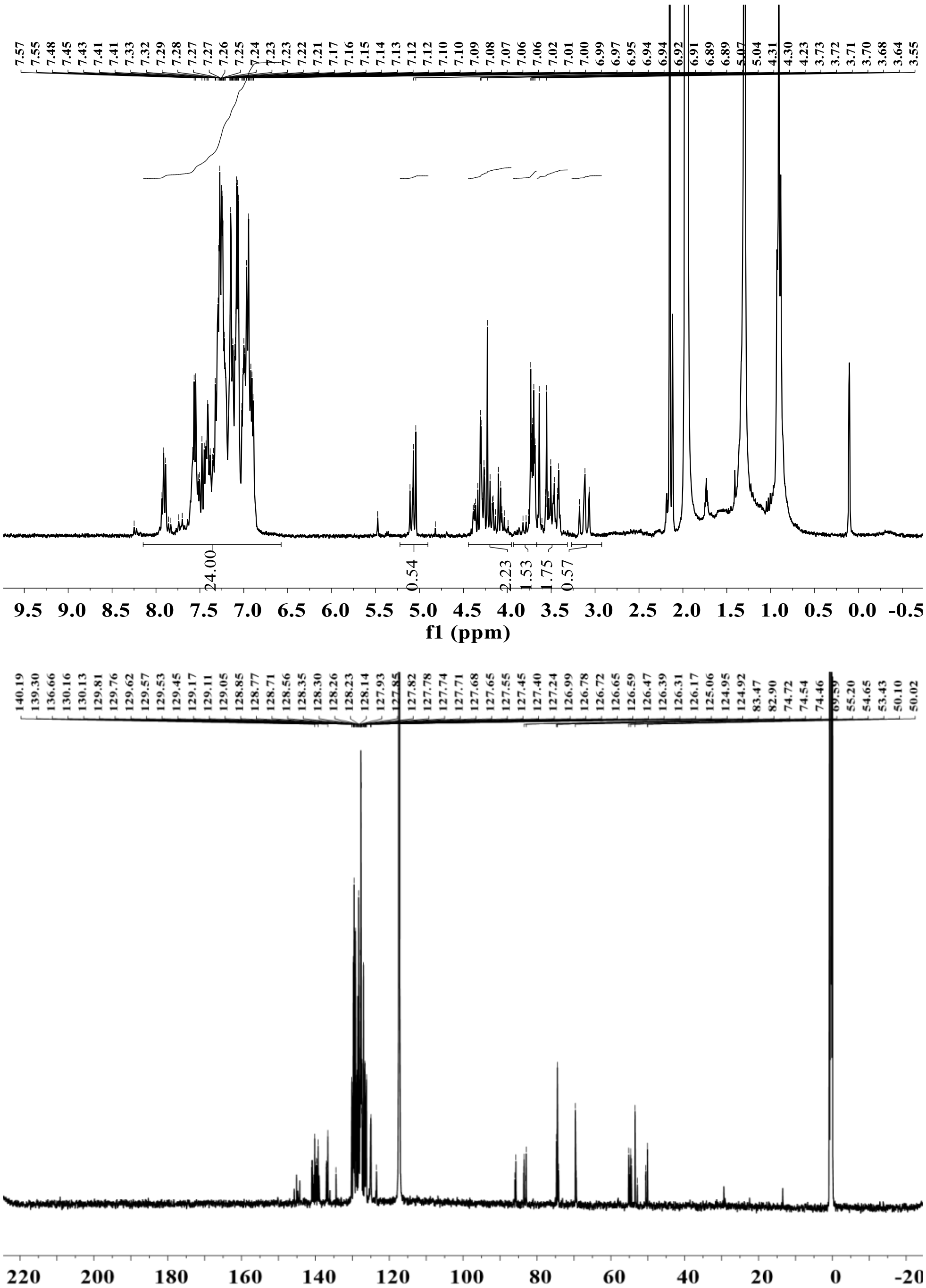
f1 (ppm)

1m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7.23
7.23
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7.13
7.12
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7.10
7.09
7.08
7.07
7.06
7.06
7.02
7.01
7.00
6.99
6.97
6.95
6.94
6.94
6.92
6.91
6.89
6.89
5.07
5.04
4.31
4.30
4.23
3.73
3.72
3.71
3.70
3.68
3.64
3.55
24.00
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2.23
1.53
1.75
0.57
9.5
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8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)
140.19
139.30
136.66
130.16
130.13
129.81
129.76
129.62
129.57
129.53
129.45
129.17
129.11
129.05
128.85
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128.71
128.56
128.35
128.30
128.26
128.23
128.14
127.93
127.85
127.82
127.78
127.74
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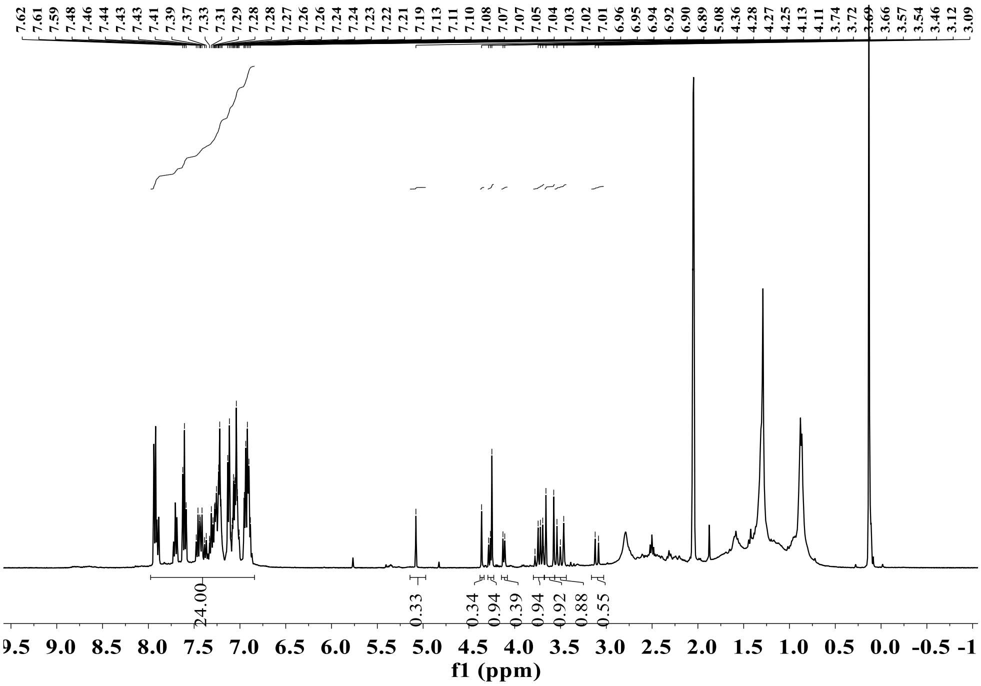
127.24
126.99
126.78
126.72
126.65
126.59
126.47
126.39
126.31
126.17
125.06
124.95
124.92
83.47
82.90
74.72
74.54
74.46
69.59
55.20
54.65
53.43
50.10
50.02
220
200
180
160
140
120
100
80
60
40
20
0
-20
f1 (ppm)

1n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7.29
7.28
7.28
7.27
7.26
7.26
7.24
7.24
7.23
7.22
7.21
7.19
7.13
7.11
7.10
7.08
7.07
7.07
7.05
7.04
7.03
7.02
7.01
6.96
6.95
6.94
6.92
6.90
6.89
5.08
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4.28
4.27
4.25
4.13
4.11
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3.72
3.69
3.66
3.57
3.54
3.46
3.12
3.09
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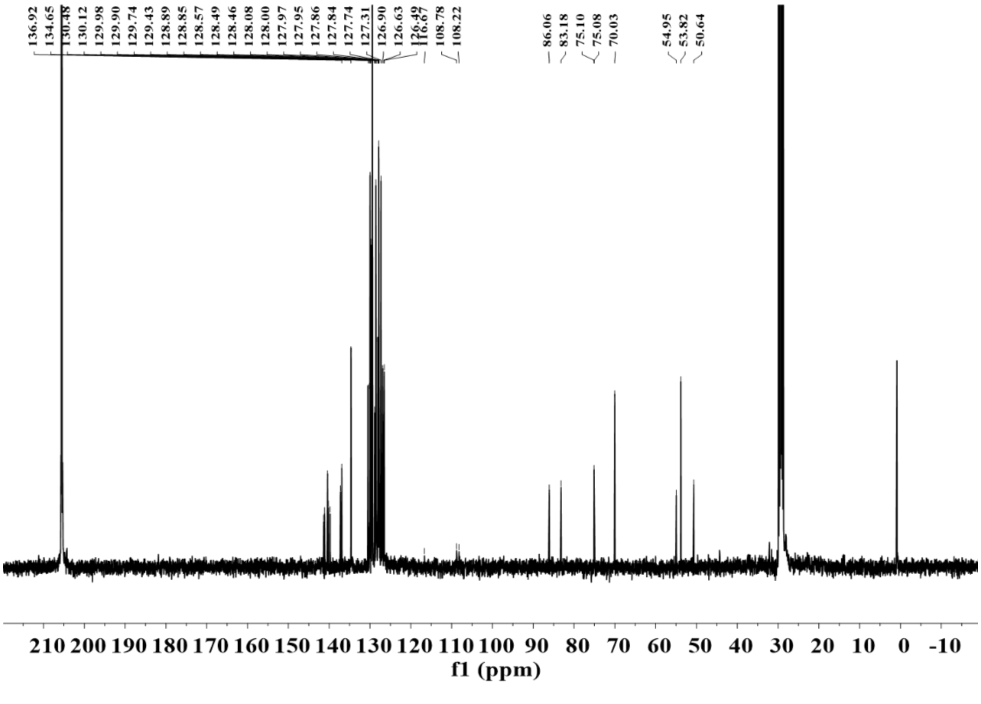
0.34
0.94
0.39
0.94
0.92
0.88
0.55
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)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128.57
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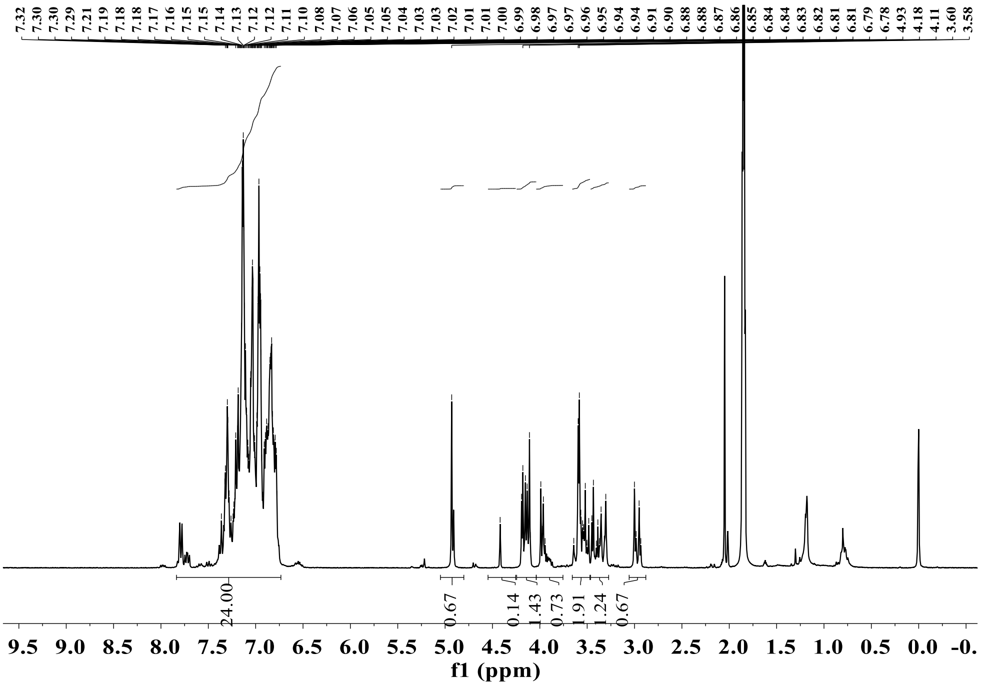
128.46
128.08
128.00
127.97
127.95
127.86
127.84
127.74
127.31
126.90
126.63
126.49
116.67
108.78
108.22
86.06
83.18
75.10
75.08
70.03
54.95
53.82
50.64
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

1o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7.14
7.13
7.12
7.12
7.11
7.10
7.08
7.07
7.06
7.05
7.05
7.04
7.03
7.03
7.02
7.01
7.01
7.00
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6.94
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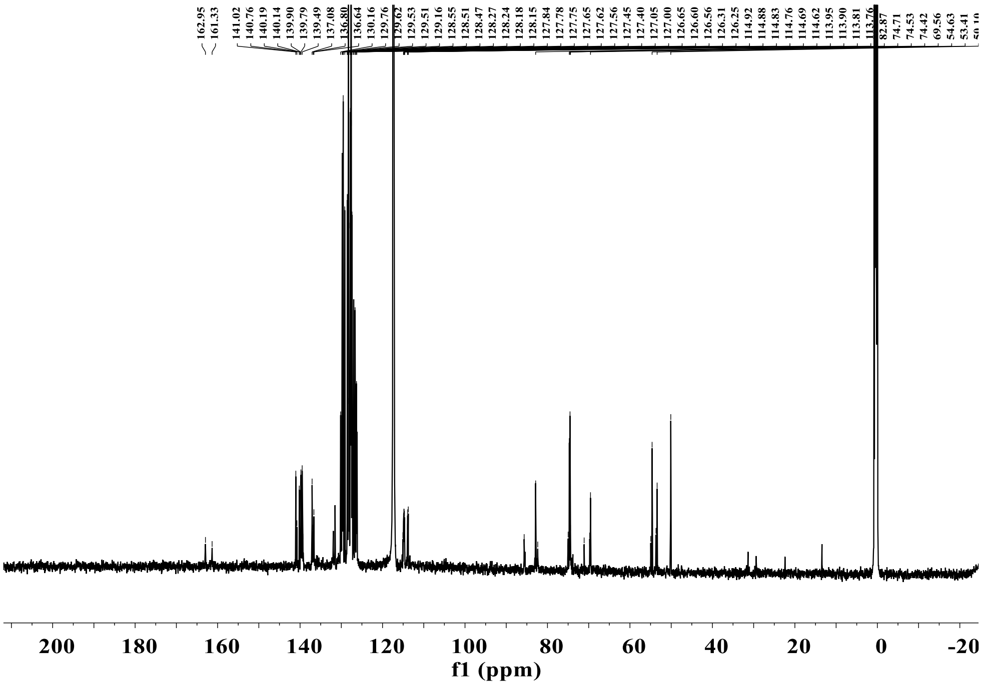
6.83
6.82
6.81
6.81
6.79
6.78
4.93
4.18
4.11
3.60
3.58
24.00
0.67
0.14
1.43
0.73
1.91
1.24
0.67
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.
f1 (ppm)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130.16
129.76
129.62
129.53
129.51
129.16
128.55
128.51
128.47
128.27
128.24
128.18
128.15
127.84
127.78
127.75
127.65
127.62
127.56
127.45
127.40
127.05
127.00
126.65
126.60
126.56
126.31
126.25
114.92
114.88
114.83
114.76
114.69
114.62
113.95
113.90
113.81
113.76
82.87
74.71
74.53
74.42
69.56
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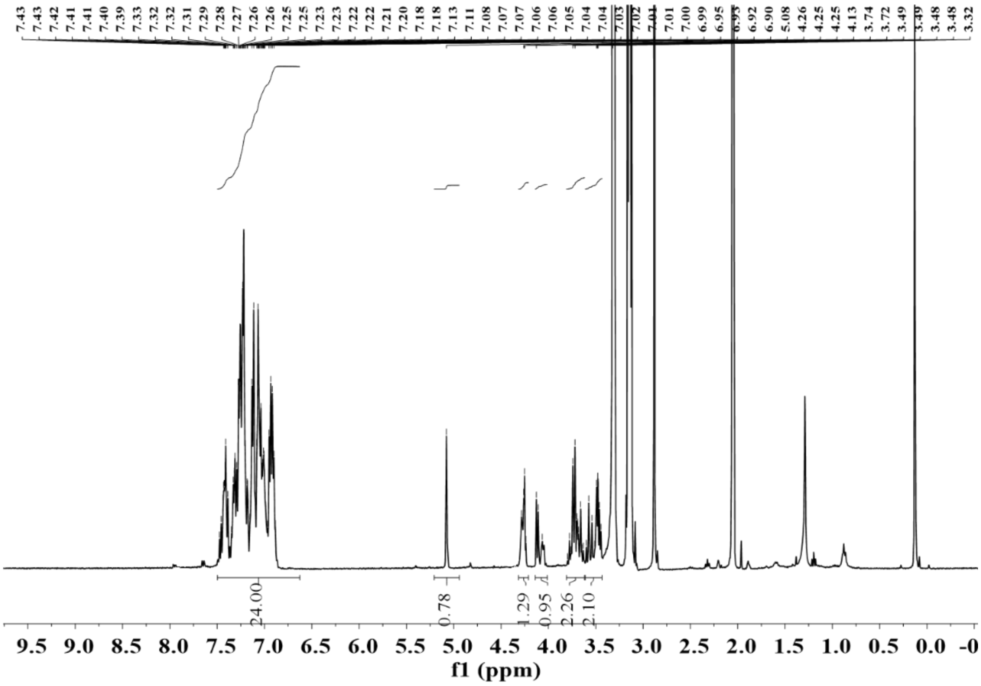
53.41
200
180
160
140
120
100
80
60
40
20
0
-20
f1 (ppm)

1p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7.23
7.22
7.22
7.21
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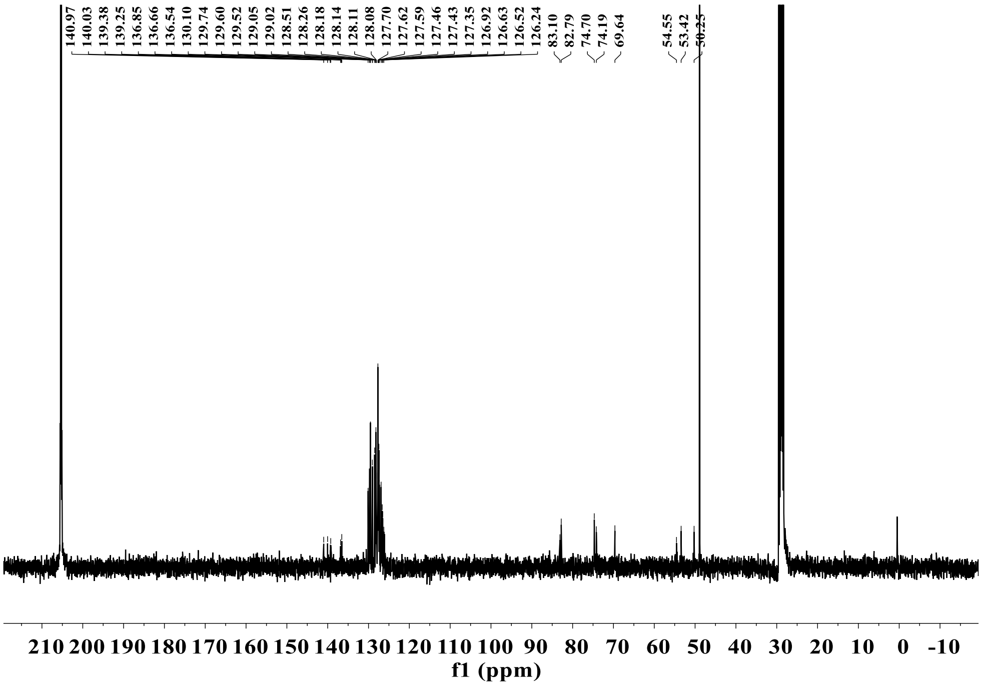
7.18
7.18
7.13
7.11
7.08
7.07
7.07
7.06
7.06
7.05
7.04
7.04
7.03
7.02
7.01
7.01
7.00
6.99
6.95
6.93
6.92
6.90
5.08
4.26
4.25
4.25
4.13
3.74
3.72
3.49
3.49
3.48
3.48
3.32
24.00
0.78
1.29
0.95
2.26
2.10
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)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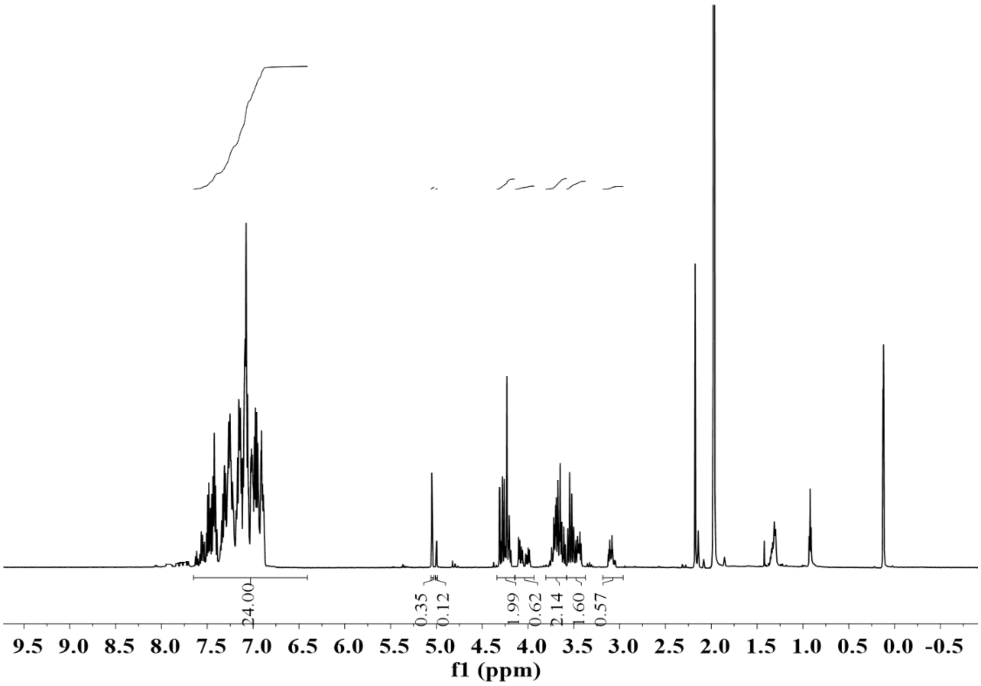
129.02
128.51
128.26
128.18
128.14
128.11
128.08
127.70
127.62
127.59
127.46
127.43
127.35
126.92
126.63
126.52
126.24
83.10
82.79
74.70
74.19
69.64
54.55
53.42
50.25
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

1q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1.99
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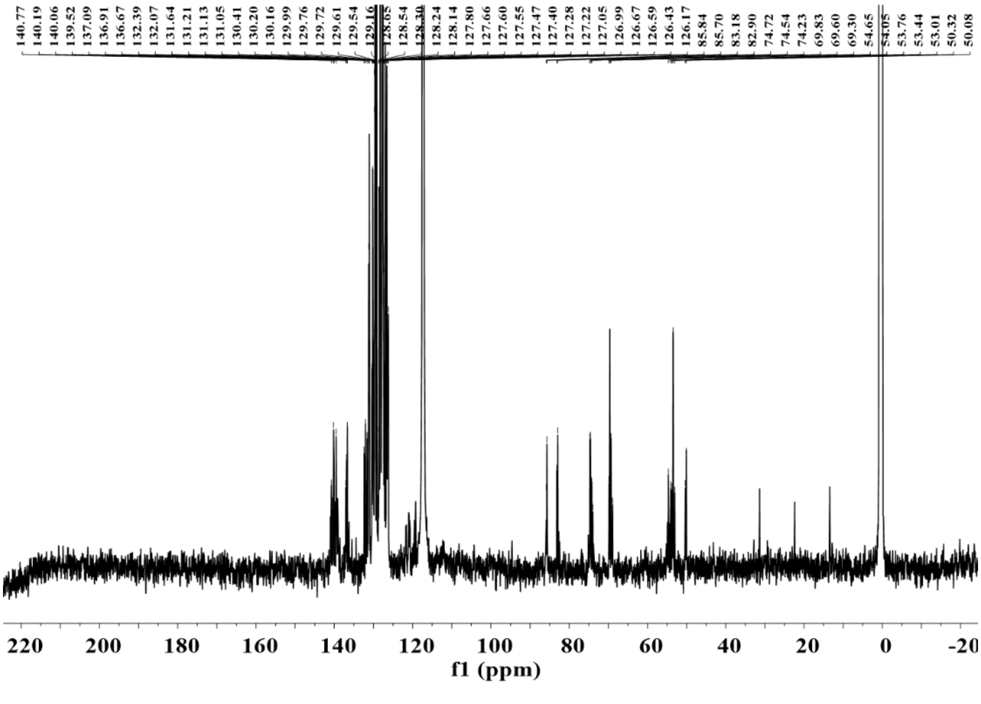
2.14
1.60
0.57
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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130.20
130.16
129.99
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129.72
129.61
129.54
129.16
128.65
128.54
128.30
128.24
128.14
127.80
127.66
127.60
127.55
127.47
127.40
127.28
127.22
127.05
126.99
126.67
126.59
126.43
126.17
85.84
85.70
83.18
82.90
74.72
74.54
74.23
69.83
69.60
69.30
54.65
54.05
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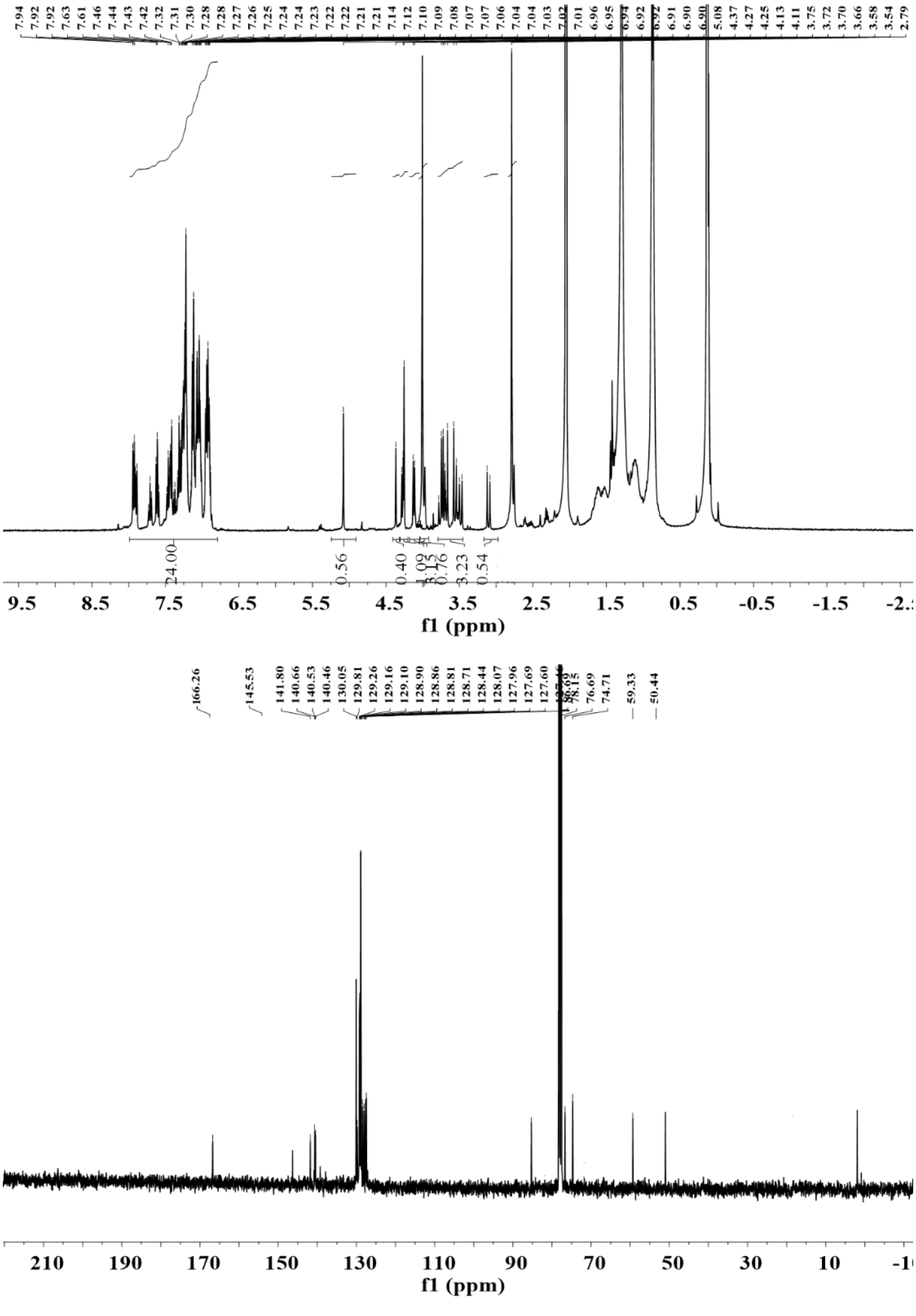
53.44
53.01
50.32
50.08
220 200 180 160 140 120 100 80 60 40 20 0 -20
f1 (ppm)

1r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7.27
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7.24
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7.23
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7.22
7.21
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7.12
7.10
7.09
7.08
7.07
7.07
7.06
7.04
7.03
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4.25
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4.11
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3.58
3.54
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0.40
1.09
3.15
0.76
3.23
0.54
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.5
f1 (ppm)
166.26
145.53
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140.53
140.46
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128.90
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128.71
128.44
128.07
127.96
127.69
127.60
78.15
76.69
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210
190
170
150
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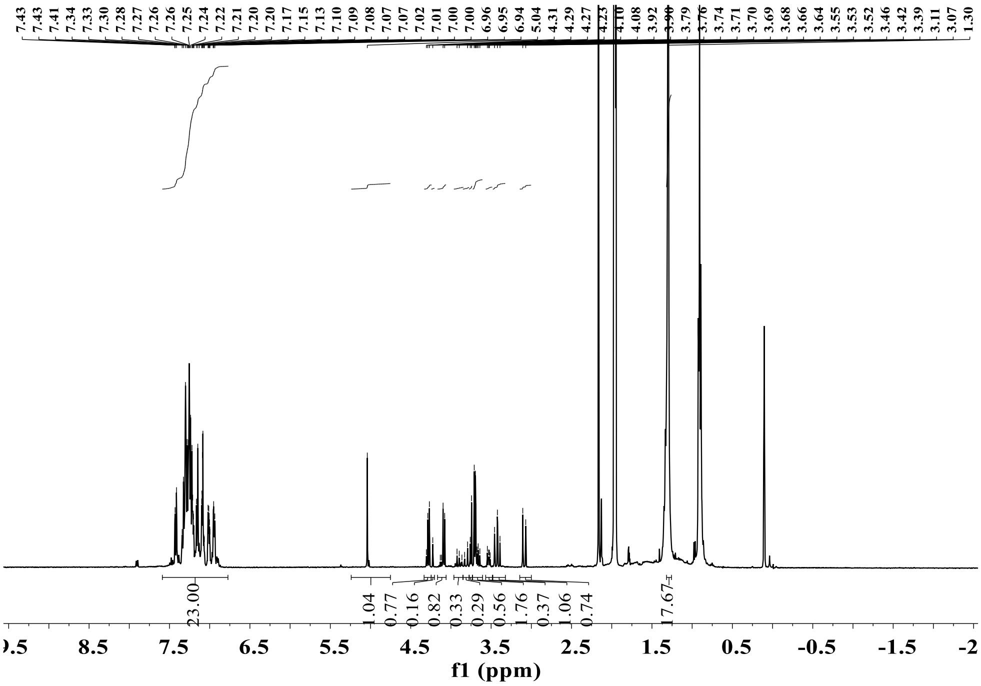
110
90
70
50
30
10
f1 (ppm)

1s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7.21
7.20
7.20
7.17
7.15
7.13
7.10
7.09
7.08
7.07
7.07
7.02
7.01
7.00
7.00
6.96
6.95
6.94
5.04
4.31
4.29
4.27
4.23
4.10
4.08
3.92
3.90
3.79
3.76
3.74
3.71
3.70
3.69
3.68
3.66
3.64
3.55
3.53
3.52
3.46
3.42
3.39
3.11
3.07
1.30
23.00
1.04
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0.16
0.82
0.33
0.29
0.56
1.76
0.37
1.06
0.74
17.67
.5
8.5
7.5
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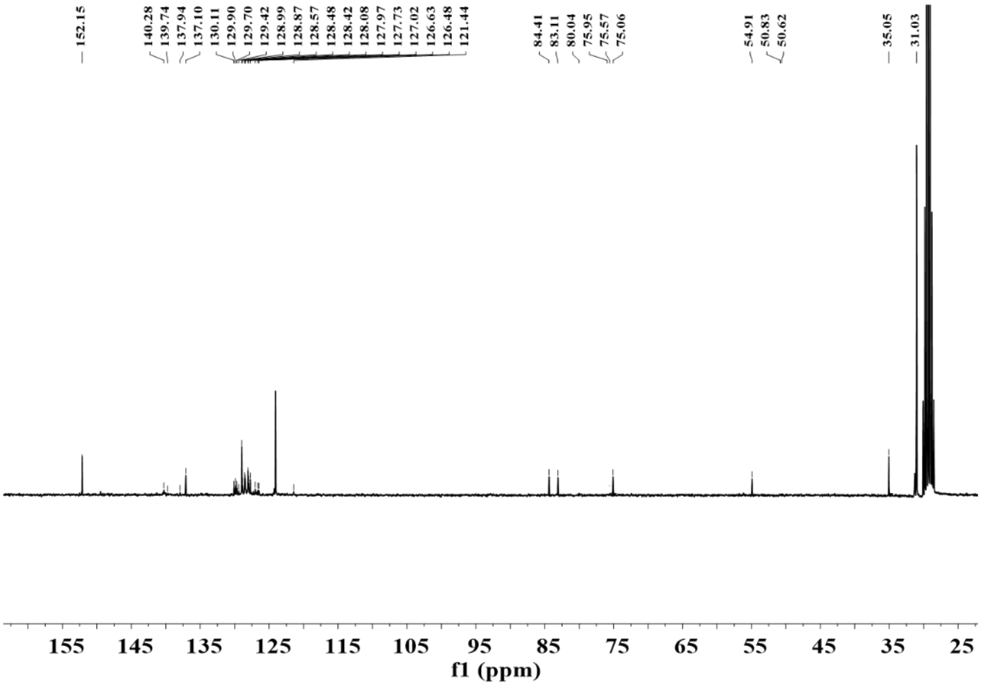
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.5
-2
f1 (ppm)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128.99
128.87
128.57
128.48
128.42
128.08
127.97
127.73
127.02
126.63
126.48
121.44
84.41
83.11
80.04
75.95
75.57
75.06
54.91
50.83
50.62
35.05
31.03
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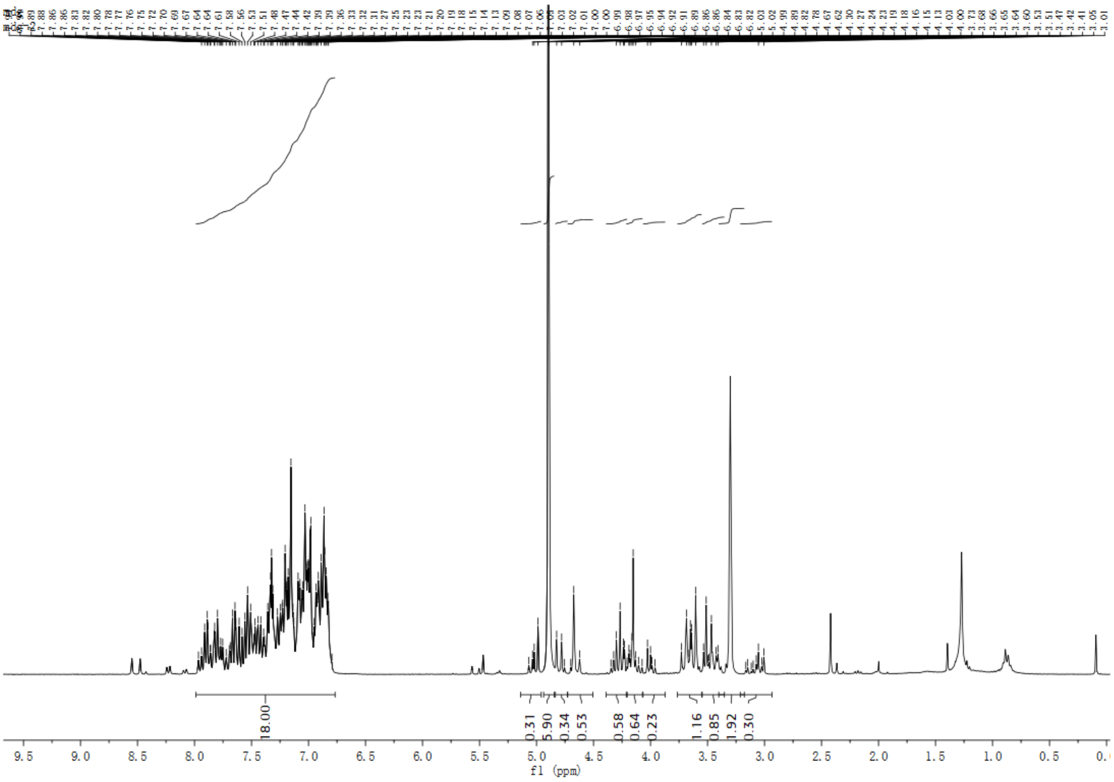
145
135
125
115
105
95
85
75
65
55
45
35
25
f1 (ppm)

1t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7.76
7.75
7.72
7.70
7.69
7.67
7.64
7.64
7.61
7.58
7.56
7.53
7.51
7.48
7.47
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7.42
7.39
7.39
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7.27
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7.02
7.01
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7.00
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6.84
6.83
6.82
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4.78
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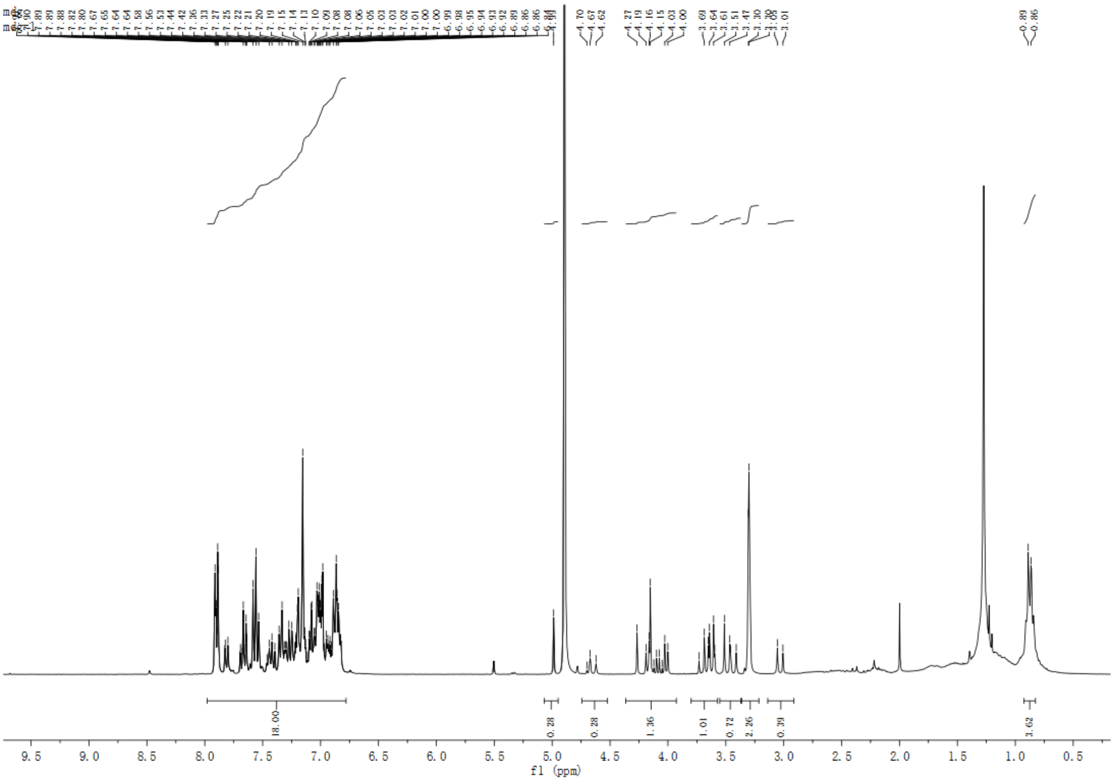
1.5
1.0
0.5
0.
f1 (ppm)

1u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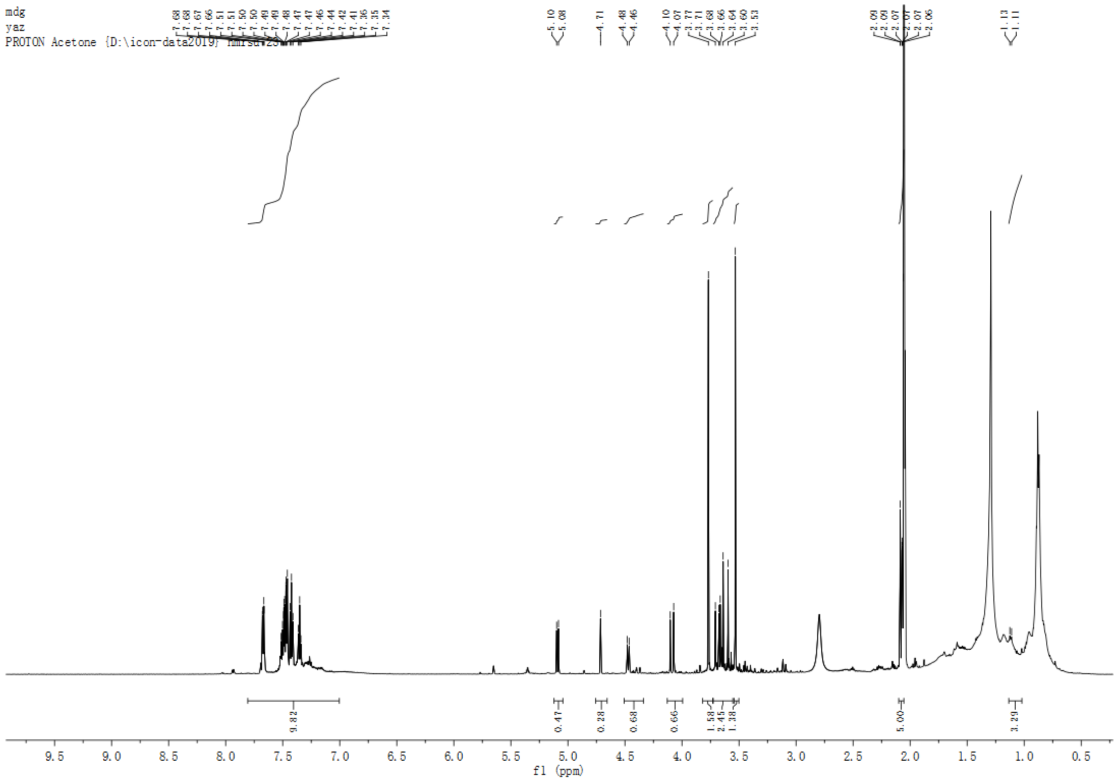
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
18.00
0.28
0.28
1.36
1.01
0.72
2.26
0.39
3.62
4.70
4.67
4.62
4.27
4.19
4.16
4.15
4.03
4.00
3.69
3.64
3.61
3.51
3.47
3.30
3.01
0.89
0.86

3c


mdg
yaz
PROTON Acetone {D:\icon-data2019} nmrsu 23
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7.51
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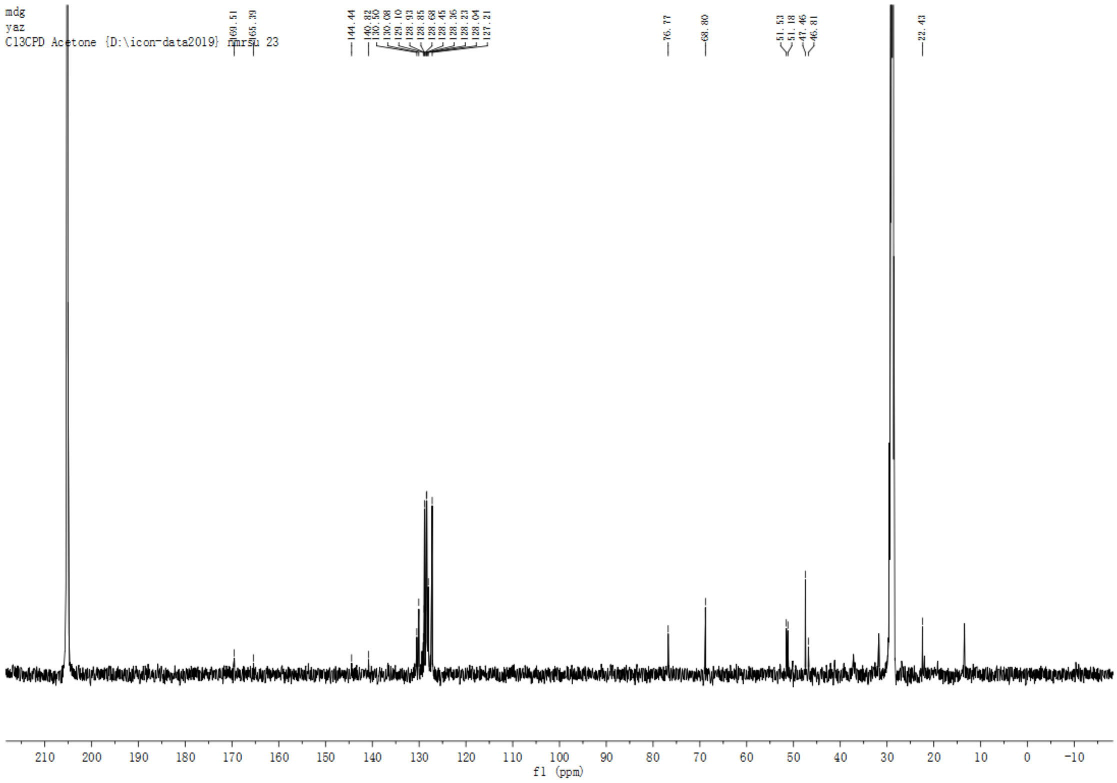
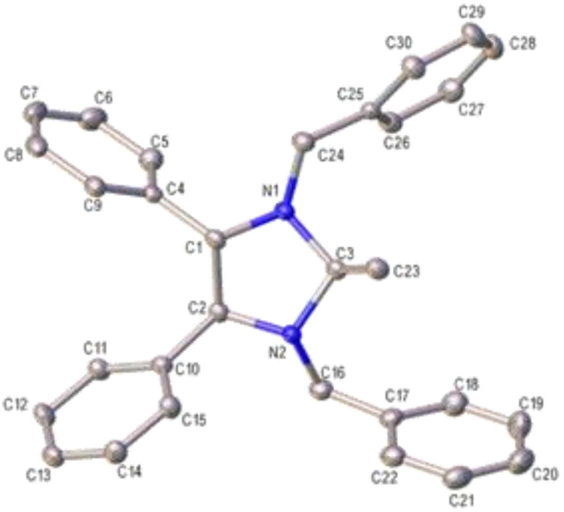
7.49
7.49
7.48
7.47
7.46
7.44
7.42
7.41
7.36
7.35
7.34
5.10
5.08
4.71
4.48
4.46
4.10
4.07
3.77
3.71
3.68
3.66
3.64
3.60
3.53
2.09
2.08
2.07
2.07
2.07
2.06
1.13
1.11
9.82
0.47
0.28
0.68
0.66
1.58
2.45
1.38
5.00
3.29
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


mdg
yaz
C13CPD Acetone {D:\icon-data2019} nmrsu 23
169.51
165.39
144.44
140.82
130.50
130.08
129.10
128.93
128.85
128.68
128.45
128.36
128.23
128.04
127.21
76.77
68.80
51.53
51.18
47.46
46.81
22.43
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

X. X-ray crystallographic data



1b 1,3-dibenzyl-2-methyl-4,5-diphenylimidazolidine

Table 1. Crystal data and structure refinement for 1b

Identification code	CCDC-1856874	
Empirical formula	$C_{30} H_{30} N_2$	
Formula weight	418.56	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 1 21/c 1	
Unit cell dimensions	a = 31.342(6) Å	α= 90°.
	b = 5.8152(12) Å	β= 115.20(3)°.
	c = 29.181(6) Å	γ = 90°.
Volume	4812(2) Å^{3}	
Z	8	
Density (calculated)	1.155 Mg/m^{3}	
Absorption coefficient	0.067 mm^{-1}	
F(000)	1792	
Theta range for data collection	0.718 to 25.200°.	
Index ranges	-37<=h<=37, -6<=k<=6, -34<=l<=34	
Reflections collected	30619	
Independent reflections	8616 [R(int) = 0.0786]	
Completeness to theta = 25.200°	99.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.0000 and 0.6336	

Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	8616 / 0 / 579
Goodness-of-fit on F^2	1.269
Final R indices [I>2sigma(I)]	R1 = 0.1039, wR2 = 0.1495
R indices (all data)	R1 = 0.1335, wR2 = 0.1605
Extinction coefficient	n/a
Largest diff. peak and hole	0.201 and -0.208 e.$Å^{-3}$