Electronic Supplementary Information

Base-mediated trimerization of enones under solvent-free and ball-milling conditions

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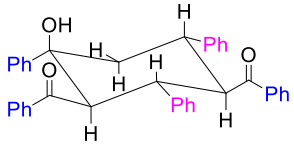
1. General information

Enones **1** were prepared by following the literature procedure.[1] Commercially available reagents were used without further purification. NMR spectra were recorded on a Bruker Advance III HD 400 NMR spectrometer (Bruker BioSpin AG, Fällanden, Switzerland; 400 MHz for ^{1}H NMR; 101 MHz for ^{13}C NMR) and a Bruker Advance III HD 500 NMR spectrometer (Bruker BioSpin AG, Fällanden, Switzerland; 500 MHz for ^{1}H NMR; 126 MHz for ^{13}C NMR). ^{1}H NMR chemical shifts were determined relative to TMS at 0.00 ppm or residual $CDCl_3$ at δ 7.26 ppm or residual DMSO-d_6 at δ 2.50 ppm. ^{13}C NMR chemical shifts were determined relative to TMS at 0.00 ppm or $CDCl_3$ at δ 77.16 ppm. Data for ^{1}H NMR and ^{13}C NMR are reported as follows: chemical shift (δ, ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet). High-resolution mass spectra (HRMS) were taken on a Waters Acquity UPLC-Xevo G2 QTof mass spectrometer (Waters, Milford, MA, USA) with FTMS-ESI in positive mode. Ball-milling reactions were performed in a Retsch MM400 mixer mill (Retsch GmbH, Haan, Germany), using a 5 mL stainless-steel jar with four 5-mm diameter stainless-steel balls and were milled at a frequency of 1800 rounds per minute (30 Hz) at room temperature.

2. Mechanosynthesis and spectral data of products 2a−2t and 2a′

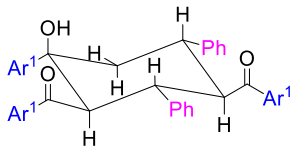
General procedure for the mechanochemical synthesis of products 2a−2t: A mixture of enone **1a** (**1b**–**1t**, 0.20 mmol) with KO^{t}Bu (0.40 mmol) and H_2O (0.60 mmol) together with four stainless-steel balls (5-mm in diameter) was introduced into a stainless-steel jar (5 mL). The reaction vessel along with another identical vessel was closed and fixed on the vibration arms of a Retsch MM400 mixer mill and vibrated at a rate of 1800 rounds per minute (30 Hz) at room temperature for 1.5 h. After completion of the reaction, the resulting mixture was combined by washing with ethyl acetate. Then, the reaction mixture was filtered through a silica gel plug with ethyl acetate as the eluent and evaporated in a vacuum. The residue was separated by flash column chromatography on silica gel with ethyl acetate/petroleum ether (2:1) as the eluent to afford product **2a** (**2b**−**2t**).



2a, Ar^{1}=Ph, Ar^{2}=Ph

Synthesis and spectral data of 2a: By following the general procedure, the reaction of **1a** (41.6 mg, 0.20 mmol) with KO^{t}Bu (44.9 mg, 0.40 mmol) and H_2O (10.0 μL, 0.60 mmol) afforded **2a** (34.0 mg, 95% yield) as a white solid.

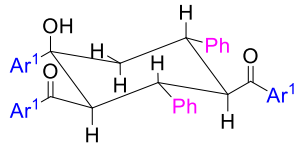
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (d, J = 7.5 Hz, 2H), 7.30–6.95 (m, 20H), 6.83 (t, J = 7.7 Hz, 2H), 6.72 (t, J = 7.4 Hz, 1H), 5.40 (d, J = 2.2 Hz, 1H), 4.49 (d, J = 11.0 Hz, 1H), 4.27–4.13 (m, 2H), 4.11–4.00 (m, 1H), 2.51 (t, J = 14.0 Hz, 1H), 2.26 (dd, J = 14.0, 3.5 Hz, 1H). Product **2a** is a known compound, and its ^{1}H NMR data are consistent with those reported in the literature.[2a, 2b]



2b, Ar^1=*p*-$CH_3OC_6H_4$, Ar^2=Ph

Synthesis and spectral data of 2b: By following the general procedure, the reaction of **1b** (47.6 mg, 0.20 mmol) with KO^{t}Bu (44.8 mg, 0.40 mmol) and H_2O (10.0 μL, 0.60 mmol) afforded **2b** (35.9 mg, 86% yield) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, J = 8.8 Hz, 2H), 7.38–7.30 (m, 4H), 7.29–7.23 (m, 2H), 7.18–7.05 (m, 4H), 6.97 (t, J = 7.3 Hz, 1H), 6.84 (t, J = 7.6 Hz, 2H), 6.75–6.67 (m, 3H), 6.56–6.49 (m, 4H), 5.56 (d, J = 2.0 Hz, 1H), 4.44–4.34 (m, 1H), 4.20–4.11 (m, 2H), 4.09–3.96 (m, 1H), 3.71 (s, 3H), 3.69 (s, 3H), 3.67 (s, 3H), 2.44 (t, J = 13.9 Hz, 1H), 2.20 (dd, J = 13.9, 3.5 Hz, 1H). Product **2b** is a known compound, and its ^{1}H NMR data are consistent with those reported in the literature.[2a]



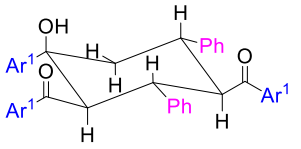
2c, Ar^1=*m*-$CH_3OC_6H_4$, Ar^2=Ph

Synthesis and spectral data of 2c: By following the general procedure, the reaction of **1c** (47.7 mg, 0.20 mmol) with KO^{t}Bu (44.6 mg, 0.40 mmol) and H_2O (10.0 μL, 0.60 mmol) afforded **2c** (19.6 mg, 47% yield) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 (d, J = 9.7 Hz, 2H), 7.18–7.07 (m, 7H), 7.03–6.90 (m, 3H), 6.89–6.69 (m, 8H), 6.66 (s, 1H), 6.63–6.56 (m, 1H), 5.36 (d, J = 1.6 Hz, 1H), 4.49–4.41 (m, 1H), 4.22–4.10 (m, 2H), 4.08–3.96 (m, 1H), 3.70 (s, 3H), 3.62 (s, 3H), 3.60 (s, 3H), 2.48 (t, J = 14.0 Hz, 1H), 2.25 (dd, J = 14.0, 3.4 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 207.2, 203.8, 159.6, 159.0, 158.9, 147.8, 142.1, 140.6, 139.8, 138.6, 129.3, 128.8, 128.6, 128.5, 128.2, 128.1, 127.1, 126.9, 120.7, 120.1, 119.6, 118.7, 117.3, 112.5, 111.7, 111.4, 111.1, 75.5, 57.2, 57.0, 55.34, 55.31, 48.3, 45.9, 43.5.

HRMS (ESI) *m/z*: Calcd for $C_{41}H_{38}O_6Na$ $[M+Na]^+$ 649.2561; found 649.2569.

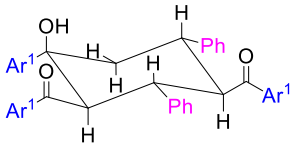


2d, Ar^1=*p*-ClC_6H_4, Ar^2=Ph

Synthesis and spectral data of 2d: By following the general procedure, the reaction of **1d** (48.7 mg, 0.20 mmol) with KO^{t}Bu (44.9 mg, 0.40 mmol) and H_2O (10.0 μL, 0.60 mmol) afforded **2d** (37.9 mg, 89% yield) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (d, J = 8.6 Hz, 2H), 7.28–6.98 (m, 17H), 6.88 (t, J = 7.7 Hz, 2H), 6.78 (t, J = 7.3 Hz, 1H), 5.37 (d, J = 2.3 Hz, 1H), 4.42–4.33 (m, 1H), 4.19–4.09 (m, 2H), 4.06–3.95 (m, 1H), 2.42 (t, J = 14.0 Hz, 1H), 2.22 (dd, J = 14.0, 3.5

Hz, 1H). Product **2d** is a known compound, and its ^{1}H NMR data are consistent with those reported in the literature.[2a, 2b]



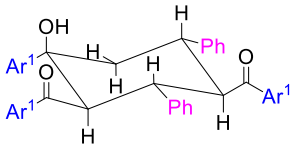
2e, Ar^1=*m*-ClC_6H_4, Ar^2=Ph

Synthesis and spectral data of 2e: By following the general procedure, the reaction of **1e** (48.1 mg, 0.20 mmol) with KO^{t}Bu (44.7 mg, 0.40 mmol) and H_2O (10.0 μL, 0.60 mmol) afforded **2e** (38.3 mg, 90% yield) as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.55 (t, J = 1.8 Hz, 1H), 7.41 (d, J = 8.0 Hz, 1H), 7.29–6.99 (m, 17H), 6.96 (t, J = 7.8 Hz, 1H), 6.92 (t, J = 7.8 Hz, 1H), 6.80 (t, J = 7.4 Hz, 1H), 5.21 (d, J = 2.4 Hz, 1H), 4.39 (d, J = 11.1 Hz, 1H), 4.21–4.08 (m, 2H), 4.03–3.95 (m, 1H), 2.46 (t, J = 14.1 Hz, 1H), 2.25 (dd, J = 14.1, 3.6 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 205.5, 202.2, 147.8, 141.4, 140.2, 139.2, 138.1, 134.4, 134.1, 133.8, 132.9, 131.8, 129.7, 129.2, 129.0, 128.5, 128.4, 127.8, 127.7, 127.44, 127.42, 127.39, 127.1, 125.7, 125.31, 125.29, 122.9, 75.1, 56.82, 56.80, 47.8, 45.4, 43.3.

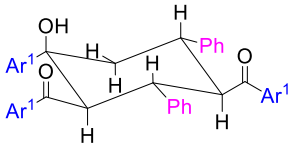
HRMS (ESI) *m/z*: Calcd for $C_{38}H_{29}{}^{35}Cl_3O_3Na$ $[M+Na]^+$ 661.1074; found 661.1073.



2f, Ar^1=*p*-BrC_6H_4, Ar^2=Ph

Synthesis and spectral data of 2f: By following the general procedure, the reaction of **1f** (57.8 mg, 0.20 mmol) with KO^{t}Bu (44.9 mg, 0.40 mmol) and H_2O (10.0 μL, 0.60 mmol) afforded **2f** (36.4 mg, 71% yield) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (d, J = 8.6 Hz, 2H), 7.32 (d, J = 8.6 Hz, 2H), 7.25–7.21 (m, 4H), 7.20–7.05 (m, 10H), 7.02 (t, J = 7.3 Hz, 1H), 6.88 (t, J = 7.7 Hz, 2H), 6.79 (t, J = 7.3 Hz, 1H), 5.36 (d, J = 2.2 Hz, 1H), 4.41–4.32 (m, 1H), 4.19–4.08 (m, 2H), 4.05–3.95 (m, 1H), 2.42 (t, J = 14.0 Hz, 1H), 2.22 (dd, J = 14.0, 3.6 Hz, 1H). Product **2f** is a known compound, and its ^{1}H NMR data are consistent with those reported in the literature.[2a, 2b]

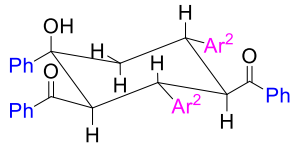


2g, Ar^1=2-thienyl, Ar^2=Ph

Synthesis and spectral data of 2g: By following the general procedure, the reaction of **1g** (42.8 mg, 0.20 mmol) with KO^{t}Bu (44.7 mg, 0.40 mmol) and H_2O (10.0 μL, 0.60 mmol) afforded **2g** (29.6 mg, 80% yield) as a white solid.

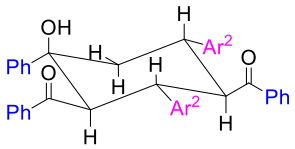
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (d, J = 4.8 Hz, 1H), 7.32 (d, J = 3.8 Hz, 1H), 7.30–

7.28 (m, 2H), 7.24–7.17 (m, 2H), 7.13 (t, J = 7.5 Hz, 3H), 7.03 (d, J = 5.2 Hz, 2H), 6.98 (d, J = 3.5 Hz, 1H), 6.94 (t, J = 7.6 Hz, 2H), 6.84–6.74 (m, 5H), 5.77–5.67 (m, 1H), 4.24–4.12 (m, 2H), 4.07–3.90 (m, 2H), 2.50–2.32 (m, 2H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 196.4, 150.5, 143.5, 134.0, 132.3, 130.5, 127.2, 127.1, 126.8, 126.4, 126.1, 126.0, 125.7, 125.6, 122.8, 121.7, 73.5, 58.5, 46.2, 41.8, 28.6.
HRMS (ESI) *m/z*: Calcd for $C_{32}H_{26}O_3S_3Na$ $[M+Na]^+$ 577.0937; found 577.0945.



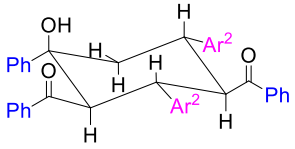
2h, Ar^1=Ph, Ar^2=*p*-$CH_3OC_6H_4$

Synthesis and spectral data of 2h: By following the general procedure, the reaction of **1h** (47.9 mg, 0.20 mmol) with KO^{t}Bu (44.7 mg, 0.40 mmol) and H_2O (10.0 μL, 0.60 mmol) afforded **2h** (34.6 mg, 87% yield) as a white solid.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, J = 7.5 Hz, 2H), 7.29–7.13 (m, 10H), 7.10–6.98 (m, 7H), 6.63 (d, J = 8.6 Hz, 2H), 6.36 (d, J = 8.7 Hz, 2H), 5.35 (d, J = 2.0 Hz, 1H), 4.49–4.39 (m, 1H), 4.19–4.06 (m, 2H), 4.05–3.93 (m, 1H), 3.63 (s, 3H), 3.47 (s, 3H), 2.45 (t, J = 14.0 Hz, 1H), 2.21 (dd, J = 14.0, 3.5 Hz, 1H). Product **2h** is a known compound, and its ^{1}H NMR data are consistent with those reported in the literature.[2]



2i, Ar^1=Ph, Ar^2=*p*-$CH_3C_6H_4$

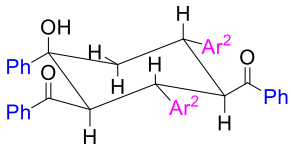
Synthesis and spectral data of 2i: By following the general procedure, the reaction of **1i** (43.8 mg, 0.20 mmol) with KO^{t}Bu (45.0 mg, 0.40 mmol) and H_2O (10.0 μL, 0.60 mmol) afforded **2i** (23.7 mg, 63% yield) as a white solid.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, J = 7.7 Hz, 2H), 7.29–7.11 (m, 10H), 7.08–6.96 (m, 7H), 6.88 (d, J = 7.7 Hz, 2H), 6.62 (d, J = 7.8 Hz, 2H), 5.35 (d, J = 1.7 Hz, 1H), 4.45 (d, J = 11.0 Hz, 1H), 4.25–4.08 (m, 2H), 4.05–3.95 (m, 1H), 2.47 (t, J = 14.1 Hz, 1H), 2.21 (dd, J = 14.1, 3.3 Hz, 1H), 2.12 (s, 3H), 1.91 (s, 3H). Product **2i** is a known compound, and its ^{1}H NMR data are consistent with those reported in the literature.[2a, 2b, 2c]



2j, Ar^1=Ph, Ar^2=*p*-ClC_6H_4

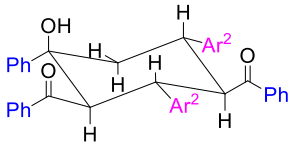
Synthesis and spectral data of 2j: By following the general procedure, the reaction of **1j** (48.0 mg, 0.20 mmol) with KO^{t}Bu (44.7 mg, 0.40 mmol) and H_2O (10.0 μL, 0.60 mmol) afforded **2j** (31.8 mg, 79% yield) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, J = 7.7 Hz, 2H), 7.32–7.15 (m, 10H), 7.14–7.02 (m, 9H), 6.80 (d, J = 8.4 Hz, 2H), 5.34 (d, J = 1.8 Hz, 1H), 4.48–4.40 (m, 1H), 4.21–4.09 (m, 2H), 4.08–3.97 (m, 1H), 2.45 (t, J = 14.0 Hz, 1H), 2.22 (dd, J = 14.0, 3.3 Hz, 1H). Product **2j** is a known compound, and its ^{1}H NMR data are consistent with those reported in the literature.[2a, 2b]



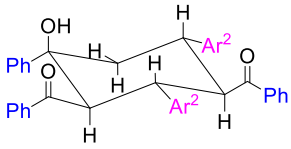
2k, Ar^1=Ph, Ar^2=*p*-BrC_6H_4

Synthesis and spectral data of 2k: By following the general procedure, the reaction of **1k** (56.8 mg, 0.20 mmol) with KO^{t}Bu (44.9 mg, 0.40 mmol) and H_2O (10.0 μL, 0.60 mmol) afforded **2k** (40.6 mg, 88% yield) as a white solid.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, J = 7.5 Hz, 2H), 7.31–7.16 (m, 10H), 7.16–7.04 (m, 7H), 7.04–6.98 (m, 2H), 7.04–6.92 (m, 2H), 5.32 (d, J = 2.0 Hz, 1H), 4.48–4.40 (m, 1H), 4.20–4.08 (m, 2H), 4.07–3.96 (m, 1H), 2.45 (t, J = 14.0 Hz, 1H), 2.22 (dd, J = 14.0, 3.4 Hz, 1H). Product **2k** is a known compound, and its ^{1}H NMR data are consistent with those reported in the literature.[2a]



2l, Ar^1=Ph, Ar^2=*m*-BrC_6H_4

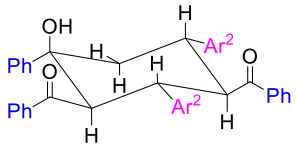
Synthesis and spectral data of 2l: By following the general procedure, the reaction of **1l** (57.6 mg, 0.20 mmol) with KO^{t}Bu (44.7 mg, 0.40 mmol) and H_2O (10.0 μL, 0.60 mmol) afforded **2l** (36.4 mg, 79% yield) as a white solid.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (d, J = 8.2 Hz, 2H), 7.47 (s, 1H), 7.40–7.23 (m, 6H), 7.21 (t, J = 7.5 Hz, 2H), 7.17–6.97 (m, 8H), 6.93 (t, J = 7.8 Hz, 1H), 6.83 (d, J = 8.0 Hz, 2H), 6.69 (t, J = 7.8 Hz, 1H), 5.39 (d, J = 2.0 Hz, 1H), 4.46 (d, J = 11.0 Hz, 1H), 4.17 (t, J = 10.9 Hz, 1H), 2.43 (t, J = 11.0 Hz, 1H), 4.05–3.95 (m, 1H), 2.43 (t, J = 14.0 Hz, 1H), 2.24 (dd, J = 14.0, 3.5 Hz, 1H). Product **2l** is a known compound, and its ^{1}H NMR data are consistent with those reported in the literature.[2a]



2m, Ar^1=Ph, Ar^2=2-naphthyl

Synthesis and spectral data of 2m: By following the general procedure, the reaction of **1m** (52.4 mg, 0.20 mmol) with KO^{t}Bu (44.7 mg, 0.40 mmol) and H_2O (10.0 μL, 0.60 mmol) afforded **2m** (19.1 mg, 45% yield) as a white solid.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (s, 1H), 7.67 (d, J = 7.9 Hz, 1H), 7.64–7.58 (m,

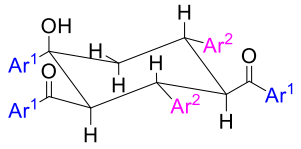
4H), 7.53 (d, J = 8.1 Hz, 1H), 7.47 (d, J = 8.4 Hz, 2H), 7.42 (d, J = 7.9 Hz, 1H), 7.38–7.30 (m, 4H), 7.13–7.01 (m, 8H), 7.12–7.01 (m, 2H), 6.96 (t, J = 7.4 Hz, 1H), 6.91 (t, J = 7.7 Hz, 2H), 6.81 (t, J = 7.6 Hz, 2H), 5.47 (d, J = 1.8 Hz, 1H), 4.67 (d, J = 11.3 Hz, 1H), 4.49 (t, J = 10.9 Hz, 1H), 4.41 (t, J = 11.2 Hz, 1H), 4.34–4.25 (m, 1H), 2.66 (t, J = 14.0 Hz, 1H), 2.35 (dd, J = 14.0, 3.4 Hz, 1H). Product **2m** is a known compound, and its ^{1}H NMR data are consistent with those reported in the literature.[2a]



2n, Ar^1=Ph, Ar^2= 2-furyl

Synthesis and spectral data of 2n: By following the general procedure, the reaction of **1n** (39.8 mg, 0.20 mmol) with KO^{t}Bu (44.9 mg, 0.40 mmol) and H_2O (10.0 μL, 0.60 mmol) afforded **2n** (21.7 mg, 63% yield) as a white solid.

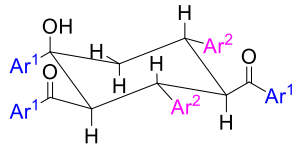
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58 (d, J = 7.7 Hz, 2H), 7.51 (d, J = 7.7 Hz, 2H), 7.45 (d, J = 7.7 Hz, 2H), 7.39–7.30 (m, 2H), 7.23 (t, J = 7.7 Hz, 2H), 7.18 (d, J = 7.2 Hz, 2H), 7.16 (d, J = 7.2 Hz, 2H), 7.09 (s, 1H), 7.05 (t, J = 7.3 Hz, 1H), 6.85 (s, 1H), 6.00 (s, 1H), 5.92 (d, J = 2.8 Hz, 1H), 5.74 (d, J = 2.8 Hz, 1H), 5.67 (s, 1H),5.37 (d, J = 2.0 Hz, 1H), 4.62 (d, J = 11.7 Hz, 1H), 4.48 (t, J = 11.3 Hz, 1H), 4.19 (t, J = 11.5 Hz, 1H), 4.09 (td, J = 12.1, 3.4 Hz, 1H), 2.45 (t, J = 14.1 Hz, 1H), 2.21 (dd, J = 14.1, 3.6 Hz, 1H). Product **2n** is a known compound, and its ^{1}H NMR data are consistent with those reported in the literature.[2b]



2o, Ar^1=*p*-$CH_3C_6H_4$, Ar^2=*p*-$CH_3OC_6H_4$

Synthesis and spectral data of 2o: By following the general procedure, the reaction of **1o** (50.1 mg, 0.20 mmol) with KO^{t}Bu (44.4 mg, 0.40 mmol) and H_2O (10.0 μL, 0.60 mmol) afforded **2o** (32.3 mg, 76% yield) as a white solid.

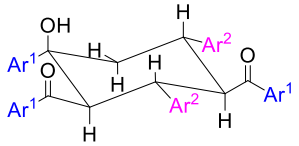
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 (d, J = 8.2 Hz, 2H), 7.24–7.18 (m, 4H), 7.16 (d, J = 8.7 Hz, 2H), 7.06–6.99 (m, 2H), 6.97 (d, J = 8.1 Hz, 2H), 6.88–6.82 (m, 4H), 6.62 (d, J = 8.7 Hz, 2H), 6.35 (d, J = 8.9 Hz, 2H), 5.42 (d, J = 2.2 Hz, 1H), 4.43–4.35 (m, 1H), 4.13–4.05 (m, 2H), 4.02–3.92 (m, 1H), 3.63 (s, 3H), 3.47 (s, 3H), 2.39 (t, J = 13.2 Hz, 1H), 2.203 (s, 3H), 2.196 (s, 3H), 2.16 (s, 3H), 2.18–2.14 (m, 1H). Product **2o** is a known compound, and its ^{1}H NMR data are consistent with those reported in the literature.[2c]



2p, Ar^1=*p*-$CH_3C_6H_4$, Ar^2=*p*-$CH_3C_6H_4$

Synthesis and spectral data of 2p: By following the general procedure, the reaction of **1p** (47.9 mg, 0.20 mmol) with KO^{t}Bu (44.5 mg, 0.40 mmol) and H_2O (10.0 μL, 0.60 mmol) afforded **2p** (30.3 mg, 75% yield) as a white solid.

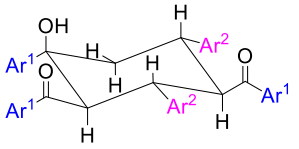
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 (d, J = 8.0 Hz, 2H), 7.20 (t, J = 7.5 Hz, 4H), 7.13 (d, J = 7.9 Hz, 2H), 7.06–6.98 (m, 2H), 6.95 (d, J = 8.0 Hz, 2H), 6.87 (d, J = 7.8 Hz, 2H), 6.83 (d, J = 7.8 Hz, 4H), 6.60 (d, J = 7.9 Hz, 2H), 5.43 (d, J = 1.7 Hz, 1H), 4.41 (d, J = 10.9 Hz, 1H), 4.19–4.08 (m, 2H), 4.04–3.92 (m, 1H), 2.42 (t, J = 13.8 Hz, 1H), 2.19 (s, 6H), 2.17–2.13 (m, 4H), 2.11 (s, 3H), 1.90 (s, 3H). Product **2p** is a known compound, and its ^{1}H NMR data are consistent with those reported in the literature.[2b, 2c]



2q, Ar^1=*p*-ClC_6H_4, Ar^2=*p*-ClC_6H_4

Synthesis and spectral data of 2q: By following the general procedure, the reaction of **1q** (55.6 mg, 0.20 mmol) with KO^{t}Bu (44.8 mg, 0.40 mmol) and H_2O (10.0 μL, 0.60 mmol) afforded **2q** (29.7 mg, 63% yield) as a white solid.

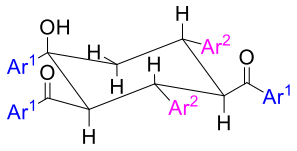
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 (d, J = 8.6 Hz, 2H), 7.22 (t, J = 8.9 Hz, 4H), 7.17 (t, J = 8.3 Hz, 4H), 7.13–6.98 (m, 8H), 6.87 (d, J = 8.7 Hz, 2H), 5.27 (d, J = 2.3 Hz, 1H), 4.33 (d, J = 11.4 Hz, 1H), 4.12 (t, J = 11.1 Hz, 1H), 4.05 (t, J = 10.8 Hz, 1H), 4.02–3.94 (m, 1H), 2.36 (t, J = 14.0 Hz, 1H), 2.18 (dd, J = 14.0, 3.4 Hz, 1H). Product **2q** is a known compound, and its ^{1}H NMR data are consistent with those reported in the literature.[2a,2b]



2r, Ar^1=*p*-ClC_6H_4, Ar^2=*p*-BrC_6H_4

Synthesis and spectral data of 2r: By following the general procedure, the reaction of **1r** (64.0 mg, 0.20 mmol) with KO^{t}Bu (44.8 mg, 0.40 mmol) and H_2O (10.0 μL, 0.60 mmol) afforded **2r** (38.1 mg, 72% yield) as a white solid.

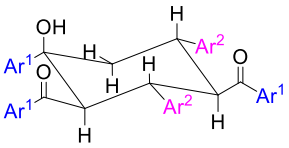
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, J = 8.6 Hz, 2H), 7.30–7.15 (m, 8H), 7.14–7.06 (m, 6H), 7.06–6.93 (m, 4H), 5.26 (d, J = 2.0 Hz, 1H), 4.33 (d, J = 11.1 Hz, 1H), 4.20–3.91 (m, 3H), 4.11 (t, J = 10.8 Hz, 1H), 4.04 (t, J = 10.6 Hz, 1H), 4.01–3.92 (m, 1H), 2.35 (t, J = 14.0 Hz, 1H), 2.18 (dd, J = 14.0, 3.3 Hz, 1H). Product **2r** is a known compound, and its ^{1}H NMR data are consistent with those reported in the literature.[2a,2c]



2s, Ar^1=*p*-$CH_3C_6H_4$, Ar^2=*p*-BrC_6H_4

Synthesis and spectral data of 2s: By following the general procedure, the reaction of **1s** (60.1 mg, 0.20 mmol) with KO^{t}Bu (44.9 mg, 0.40 mmol) and H_2O (10.0 μL, 0.60 mmol) afforded **2s** (43.6 mg, 89% yield) as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 (d, J = 8.2 Hz, 2H), 7.24–7.17 (m, 6H), 7.12 (d, J = 8.5 Hz, 2H), 6.98 (d, J = 8.0 Hz, 4H), 6.94 (d, J = 8.8 Hz, 2H), 6.91–6.85 (m, 4H), 5.41 (d, J = 2.4 Hz, 1H), 4.39 (d, J = 11.1 Hz, 1H), 4.17–4.05 (m, 2H), 4.03–3.95 (m, 1H), 2.39 (t, J = 13.9 Hz, 1H), 2.23 (s, 3H), 2.22 (s, 3H), 2.20–2.17 (m, 1H), 2.16 (s, 3H). Product **2s** is a known compound, and its ^{1}H NMR data are consistent with those reported in the literature.[2c]

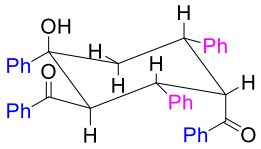


2t, Ar^1=*p*-ClC_6H_4, Ar^2=*p*-$CH_3C_6H_4$

Synthesis and spectral data of 2t: By following the general procedure, the reaction of **1t** (51.5 mg, 0.20 mmol) with KO^{t}Bu (44.8 mg, 0.40 mmol) and H_2O (10.0 μL, 0.60 mmol) afforded **2t** (32.4 mg, 73% yield) as a white solid.

^{1}H NMR (500 MHz, DMSO-d_6) δ 7.59 (d, J = 8.6 Hz, 2H), 7.37 (t, J = 8.1 Hz, 4H), 7.23 (d, J = 8.5 Hz, 5H), 7.19 (d, J = 7.9 Hz, 2H), 7.14 (d, J = 8.7 Hz, 3H), 6.91 (d, J = 7.9 Hz, 2H), 6.68 (d, J = 8.0 Hz, 2H), 5.29 (s, 1H), 4.91 (d, J = 11.6 Hz, 1H), 4.65 (t, J = 11.1 Hz, 1H), 3.90 (t, J = 11.3 Hz, 1H), 3.68–3.59 (m, 1H), 2.83 (t, J = 13.0 Hz, 1H), 2.08 (s, 3H), 1.89 (s, 3H), 1.78 (dd, J = 13.4, 3.3 Hz, 1H). Product **2t** is a known compound, and its ^{1}H NMR data are consistent with those reported in the literature.[2c]

Mechanochemical synthesis of product 2a': A mixture of **1a** (41.8 mg, 0.20 mmol) with KO^{t}Bu (22.7 mg, 0.20 mmol) and H_2O (10.0 μL, 0.60 mmol) together with four stainless-steel balls (5-mm in diameter) was introduced into a stainless-steel jar (5 mL). The reaction vessel along with another identical vessel was closed and fixed on the vibration arms of a Retsch MM400 mixer mill and vibrated at a rate of 1800 rounds per minute (30 Hz) at room temperature for 1.5 h. After completion of the reaction, the resulting mixture was combined by washing with ethyl acetate. Then, the reaction mixture was filtered through a silica gel plug with ethyl acetate as the eluent and evaporated in a vacuum. The residue was separated by flash column chromatography on silica gel with ethyl acetate/petroleum ether (2:1) as the eluent to afford product **2a′** (20.7 mg, 58% yield) along with product **2a** (7.5 mg, 21% yield) as a white solid.



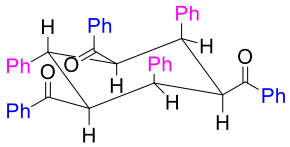
2a′, Ar^1=Ph, Ar^2=Ph

Spectral data of 2a′:

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.79 (d, J = 8.2 Hz, 2H), 7.46 (d, J = 8.0 Hz, 2H), 7.31–7.22 (m, 3H), 7.20 (d, J = 7.9 Hz, 2H), 7.14 (t, J = 7.3 Hz, 1H), 7.12–7.02 (m, 9H), 7.00–6.92 (m, 3H), 6.85–6.75 (m, 3H), 5.72 (d, J = 12.0 Hz, 1H), 5.23 (d, J = 2.3 Hz, 1H), 4.37 (t, J = 4.3 Hz, 1H), 4.25–4.10 (m, 2H), 3.42 (t, J = 13.7 Hz, 1H), 2.05 (dd, J = 13.7, 3.0 Hz, 1H). Product **2a′** is a known compound, and its ^{1}H NMR data are consistent with those reported in the literature.[3]

3. Liquid-phase reaction of 1a with KO^{t}Bu and H_2O

To a 25 mL Schlenk tube with a magnetic stir bar were added **1a** (47.1 mg, 0.20 mmol), KO^{t}Bu (44.6 mg, 0.40 mmol) and H_2O (10.0 μL, 0.60 mmol). Subsequently, diethyl ether (Et_2O, 1.5 mL) was added by syringe. The resulting mixture was stirred at room temperature for 48 h. Then, the reaction mixture was filtered through a silica gel plug with ethyl acetate as the eluent and evaporated in vacuo. The residue was separated by flash column chromatography on silica gel with ethyl acetate/petroleum ether as the eluent to afford products **2a** (8.9 mg, 22% yield), **3a** (12.7 mg, 27% yield) and other unidentified byproducts.



3a, Ar^1=Ph, Ar^2=Ph

Spectral data of 3a:

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, J = 7.2 Hz, 4H), 7.47 (t, J = 7.4 Hz, 2H), 7.40–7.28 (m, 8H), 7.22–6.93 (m, 14H), 6.85 (t, J = 7.4 Hz, 2H), 4.77 (dd, J = 12.1, 5.3 Hz, 2H), 4.45 (t, J = 11.8 Hz, 2H), 4.19 (t, J = 11.5 Hz, 1H), 3.99 (t, J = 5.2 Hz, 1H). Product **3a** is a known compound, and its ^{1}H NMR data are consistent with those reported in the literature.[2a]

Other solvents, including toluene (1.5 mL), dimethyl sulfoxide (DMSO, 1.5 mL), tetrahydrofuran (THF, 1.5 mL) and acetonitrile (CH_3CN, 1.5 mL), were also used to replace diethyl ether in the above procedure. However, all of them led to lower product yields.

4. Control experiments

4.1 Scavenging experiments with TEMPO

Scavenging experiment with 3 equiv. of TEMPO: A mixture of **1a** (41.8 mg, 0.20 mmol), KO^{t}Bu (44.9 mg, 0.40 mmol), H_2O (10.0 μL, 0.60 mmol) and 2,2,6,6-tetramethylpiperidinooxy (TEMPO, 94.2 mg, 0.60 mmol) together with four stainless-

steel balls (5-mm in diameter) was introduced into a stainless-steel jar (5 mL). The reaction vessel along with another identical vessel was closed and fixed on the vibration arms of a Retsch MM400 mixer mill and vibrated at a rate of 1800 rounds per minute (30 Hz) at room temperature for 1.5 h. After completion of the reaction, the resulting mixture was combined by washing with ethyl acetate. Then, the reaction mixture was filtered through a silica gel plug with ethyl acetate as the eluent and evaporated in vacuo. The residue was separated by flash column chromatography on silica gel with ethyl acetate/petroleum ether (2:1) as the eluent to afford product **2a** (16.9 mg, 47% yield) as a white solid.

Scavenging experiments with 10 equiv. of TEMPO: A mixture of **1a** (41.4 mg, 0.20 mmol), KO^{t}Bu (44.4 mg, 0.40 mmol), H_2O (10.0 μL, 0.60 mmol) and TEMPO (312.0 mg, 2.0 mmol) together with four stainless-steel balls (5-mm in diameter) was introduced into a stainless-steel jar (5 mL). The reaction vessel along with another identical vessel was closed and fixed on the vibration arms of a Retsch MM400 mixer mill and vibrated at a rate of 1800 rounds per minute (30 Hz) at room temperature for 1.5 h. After completion of the reaction, the resulting mixture was combined by washing with ethyl acetate. Then, the reaction mixture was filtered through a silica gel plug with ethyl acetate as the eluent and evaporated in a vacuum. The residue was separated by flash column chromatography on silica gel with ethyl acetate/petroleum ether (2:1) as the eluent to afford product **2a** (14.7 mg, 41% yield) as a white solid.

4.2 Mechanochemical reaction under an inert atmosphere

A mixture of **1a** (41.6 mg, 0.20 mmol), KO^{t}Bu (44.9 mg, 0.40 mmol) and H_2O (10.0 μL, 0.60 mmol) together with four stainless-steel balls (5-mm in diameter) was introduced into a stainless-steel jar (5 mL). The jar was filled with nitrogen (N_2) and tightened in a glovebox. The reaction vessel along with another identical vessel was closed and fixed on the vibration arms of a Retsch MM400 mixer mill and vibrated at a rate of 1800 rounds per minute (30 Hz) at room temperature for 1.5 h. After completion of the reaction, the resulting mixture was combined by washing with ethyl acetate. Then, the reaction mixture was filtered through a silica gel plug with ethyl acetate as the eluent and evaporated in a vacuum. The residue was separated by flash column chromatography on silica gel with ethyl acetate/petroleum ether (2:1) as the eluent to afford product **2a** (29.6 mg, 83% yield) as a white solid.

4.3 HRMS detection of benzaldehyde from the reaction mixture

A mixture of **1a** (41.1 mg, 0.20 mmol), KO^{t}Bu (44.6 mg, 0.40 mmol) and H_2O (10.0 μL, 0.60 mmol) together with four stainless-steel balls (5-mm in diameter) was introduced into a stainless-steel jar (5 mL). The reaction vessel along with another identical vessel was closed and fixed on the vibration arms of a Retsch MM400 mixer mill and vibrated at a rate of 1800 rounds per minute (30 Hz) at room temperature for 0.5 h. The benzaldehyde could be detected by high-resolution mass spectrometry (HRMS). HRMS (ESI) *m/z*: Calcd for C_7H_7O $[M+H]^+$ 107.0491; found 107.0487 (**Figure S1**).

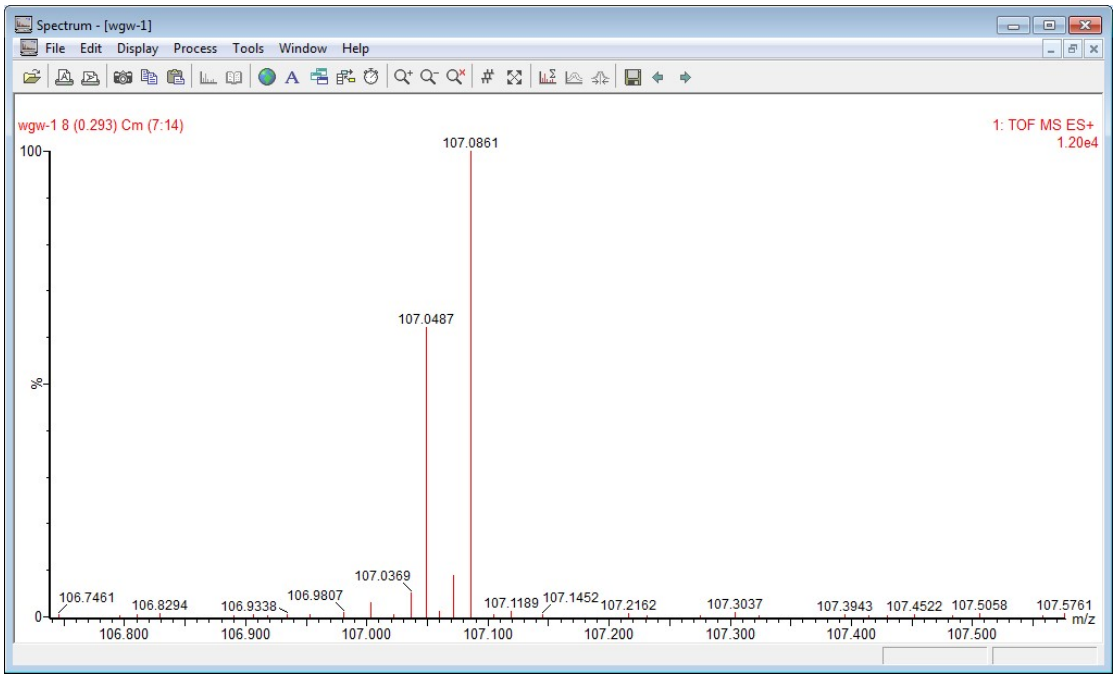
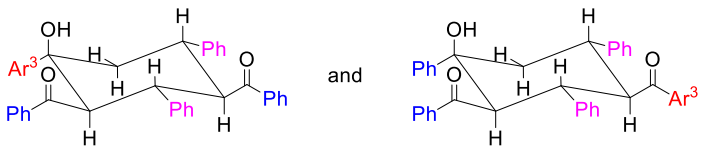


Figure S1 HRMS detection of benzaldehyde

4.4 Mechanosynthesis of products 2u and 2v

A mixture of chalcone **1a** (41.6 mg, 0.20 mmol), 4'-methoxyacetophenone (14.0 μL, 0.10 mmol), KO^{t}Bu (44.9 mg, 0.40 mmol) and H_2O (10.0 μL, 0.60 mmol) together with four stainless-steel balls (5-mm in diameter) was introduced into a stainless-steel jar (5 mL). The reaction vessel along with another identical vessel was closed and fixed on the vibration arms of a Retsch MM400 mixer mill and vibrated at a rate of 1800 rounds per minute (30 Hz) at room temperature for 1.5 h. After completion of the reaction, the resulting mixture was combined by washing with ethyl acetate. Then, the reaction mixture was filtered through a silica gel plug with ethyl acetate as the eluent and evaporated in a vacuum. The residue was separated by flash column chromatography on silica gel with ethyl acetate/petroleum ether as the eluent to afford product **2a** (13.2 mg, 37% yield) along with isomeric products **2u** and **2v** (24.1 mg, 41% yield) as inseparable white solids.



2u & **2v**, Ar^1=Ph, Ar^2=Ph, Ar^3=*p*-$CH_3OC_6H_4$

Spectral data of 2u and 2v:

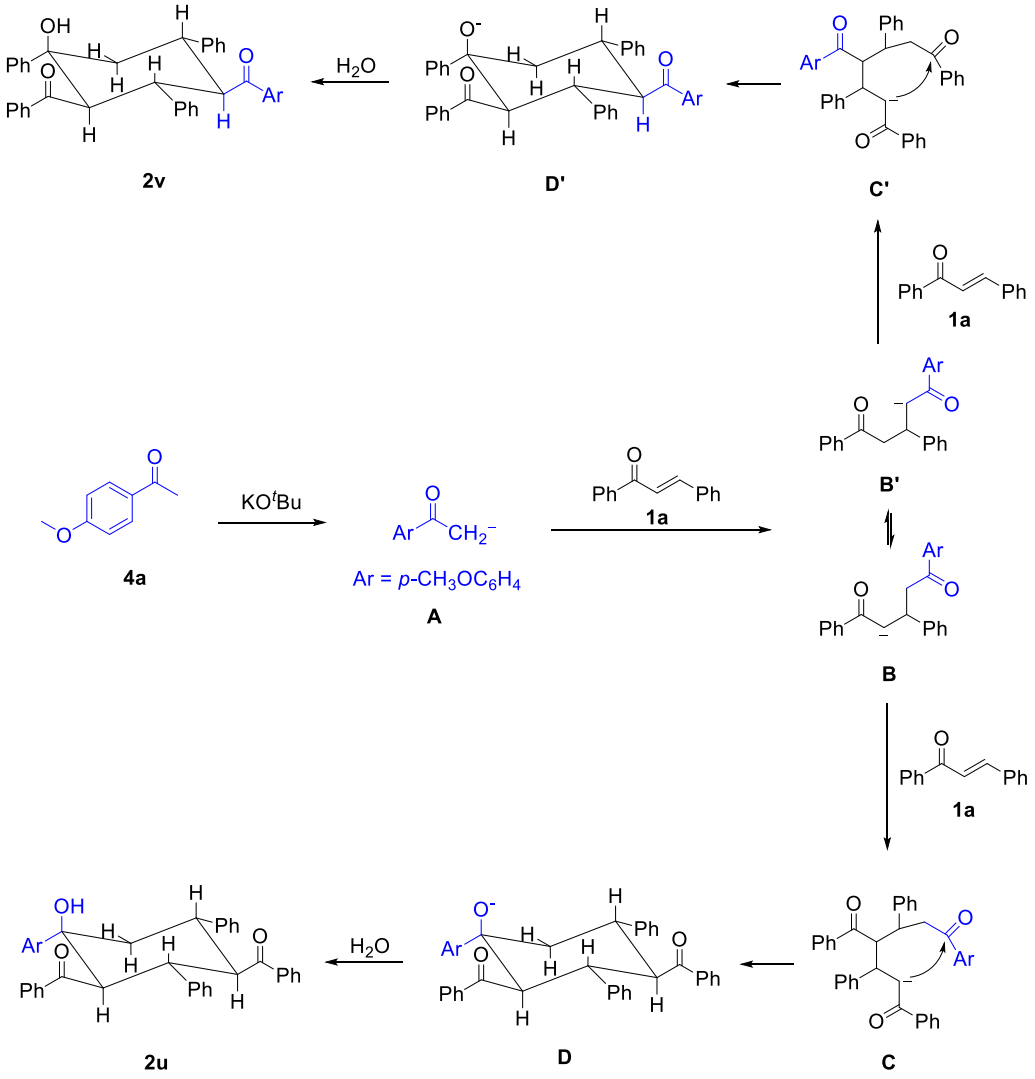
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59–7.52 (m, 2H, **2u** & **2v**), 7.33 (d, J = 8.8 Hz, 2H, **2u** & **2v**), 7.30–7.12 (m, 9H, **2u** & **2v**), 7.11–7.06 (m, 2H, **2u** & **2v**), 7.05–7.00 (m, 3H, **2u** & **2v**), 7.00–6.94 (m, 1H, **2u** & **2v**), 6.87–6.81 (m, 2H, **2u** & **2v**), 6.75–6.68 (m, 1H, **2u** & **2v**), 6.51 (d, J = 8.7 Hz, 2H, **2u** & **2v**), 5.58 (d, J = 2.1 Hz, 0.5H, **2v**), 5.40 (d, J =

2.1 Hz, 0.5H, **2u**), 4.53–4.42 (m, 1H, **2u** & **2v**), 4.26–4.15 (m, 2H, **2u** & **2v**), 4.12–4.02 (m, 1H, **2u** & **2v**), 3.70–6.66 (m, 3H, **2u** & **2v**), 2.56–4.46 (m, 1H, **2u** & **2v**), 2.29–2.21 (m, 1H, **2u** & **2v**).

^{13}C NMR (126 MHz, $CDCl_3$) δ 207.3, 204.7, 203.7, 201.4, 163.3, 162.5, 146.1, 146.0, 142.3, 142.2, 139.0, 138.9, 138.8, 138.1, 132.6, 132.3, 131.8, 131.7, 130.9, 130.4, 129.9, 128.34, 128.31, 128.2, 128.1, 128.05, 127.99, 127.9, 127.70, 127.66, 127.5, 127.4, 126.8, 126.7, 126.6, 124.82, 124.80, 112.9, 112.8, 75.4, 75.3, 56.83, 56.79, 55.7, 55.3, 55.2, 48.05, 48.01, 45.94, 45.90, 43.4, 43.3.

HRMS (ESI) *m/z*: Calcd for $C_{39}H_{34}O_4Na$ $[M+Na]^+$ 589.2349; found 589.2351.

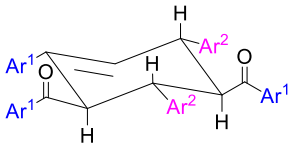
Scheme S1 Proposed reaction mechanism for the formation of **2u** and **2v**



5. Mechanochemical synthesis of products 4a and 4q

A mixture of **2a** (**2q**, 0.10 mmol) and TfOH (0.30 mmol) together with four stainless-steel balls (5-mm in diameter) was introduced into a stainless-steel jar (5 mL). The reaction vessel along with another identical vessel was closed and fixed on the vibration arms of a Retsch MM400 mixer mill and vibrated at a rate of 1800 rounds per minute

(30 Hz) at room temperature for 1.5 h. After completion of the reaction, the resulting mixture was combined by washing with ethyl acetate. Then, the reaction mixture was filtered through a silica gel plug with ethyl acetate as the eluent and evaporated in a vacuum. The residue was separated by flash column chromatography on silica gel with ethyl acetate/petroleum ether as the eluent to afford product **4a** (**4q**).



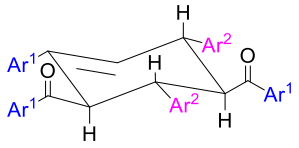
4a, Ar^1=Ph, Ar^2=Ph

Synthesis and spectral data of 4a: By following the general procedure, the reaction of **2a** (53.9 mg, 0.10 mmol) with TfOH (26.5 μL, 0.30 mmol) afforded **4a** (48.2 mg, 93% yield) as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.49 (d, J = 7.2 Hz, 2H), 7.37–6.97 (m, 20H), 6.96–6.80 (m, 3H), 6.34 (s, 1H), 5.12 (d, J = 9.2 Hz, 1H), 4.35 (d, J = 8.6 Hz, 1H), 4.24 (t, J = 10.7 Hz, 1H), 3.95 (t, J = 10.8 Hz, 1H);

^{13}C NMR (126 MHz, $CDCl_3$) δ 204.3, 201.9, 143.0, 140.4, 139.7, 139.2, 138.1, 137.8, 132.5, 132.1, 131.8, 128.8, 128.39, 128.36, 128.3, 128.2, 128.1, 127.8, 127.4, 127.3, 127.14, 127.10, 126.5, 55.7, 55.3, 49.8, 48.1.

HRMS (ESI) *m/z*: Calcd for $C_{38}H_{30}O_2Na$ $[M+Na]^+$ 541.2138; found 541.2145.



4q, Ar^1=*p*-ClC_6H_4, Ar^2=*p*-ClC_6H_4

Synthesis and spectral data of 4q: By following the general procedure, the reaction of **2q** (70.1 mg, 0.10 mmol) with TfOH (26.5 μL, 0.30 mmol) afforded **4q** (61.2 mg, 89% yield) as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45 (d, J = 8.7 Hz, 2H), 7.23–7.20 (m, 2H), 7.19–7.11 (m, 8H), 7.11–7.06 (m, 4H), 6.95 (s, 4H), 6.22 (t, J = 1.9 Hz, 1H), 4.98–4.90 (m, 1H), 4.31–4.23 (m, 1H), 4.07 (dd, J = 10.9, 10.5 Hz, 1H), 3.87 (dd, J = 11.2, 10.9 Hz, 1H). Product **4q** is a known compound, and its ^{1}H NMR data are consistent with those reported in the literature.[4]

6. X-ray data of product 2a

White flake crystals of **2a** were obtained by slow diffusion of **2a** in $CHCl_3$/*n*-hexane solution at about 4 °C. Single-crystal X-ray diffraction data were collected on a diffractometer (SuperNova, Rigaku) equipped with a CCD area detector using graphite-monochromated CuKα radiation (λ = 1.54184 Å) in the scan range of 7.222° < 2θ < 145.894°. The structure was solved with direct methods using SHELXT and refined

with full-matrix least-squares refinement using the SHELXL program within OLEX2. Crystallographic data have been deposited in the Cambridge Crystallographic Data Centre as deposition number CCDC 2277780.

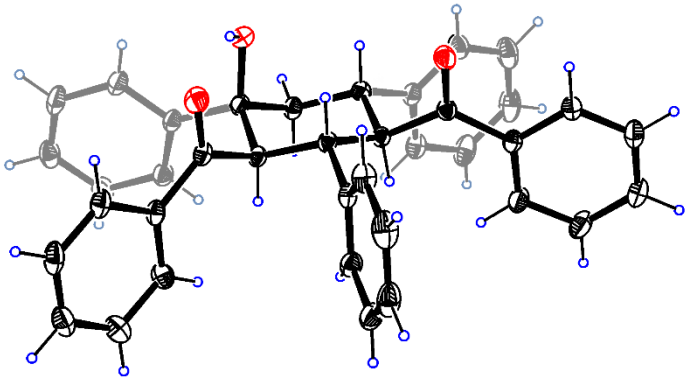


Figure S2 ORTEP diagram for one enantiomer of racemic **2a** with 15% thermal ellipsoids.

Table S1 Crystal data and structure refinement for **2a**.

Identification code	2277780
Crystal	$C_{38}H_{32}O_3$
Empirical formula	$C_{38}H_{32}O_3$
Formula weight	536.63
Temperature/K	293(2)
Crystal system	triclinic
Space group	P-1
a/Å	12.4366(4)
b/Å	12.5553(3)
c/Å	20.7421(7)
α/°	94.962(2)
β/°	97.385(3)
γ/°	101.068(2)
Volume/Å^3	3131.56(17)
Z	4
ρ_{calc} g/cm^3	1.138
μ/mm^{-1}	0.556
F(000)	1136.0
Crystal size/mm^3	0.2 × 0.18 × 0.05
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	7.222 to 145.894
Index ranges	-15 ≤ h ≤ 13, -11 ≤ k ≤ 15, -25 ≤ l ≤ 25
Reflections collected	22430
Independent reflections	12132 [R_{int} = 0.0287, R_{sigma} = 0.0366]
Data/restraints/parameters	12132/0/742
Goodness-of-fit on F^2	1.040
Final R indexes [I>=2σ (I)]	R_1 = 0.0518, wR_2 = 0.1492
Final R indexes [all data]	R_1 = 0.0660, wR_2 = 0.1626
Largest diff. peak/hole / e Å^{-3}	0.63/-0.26

7. References

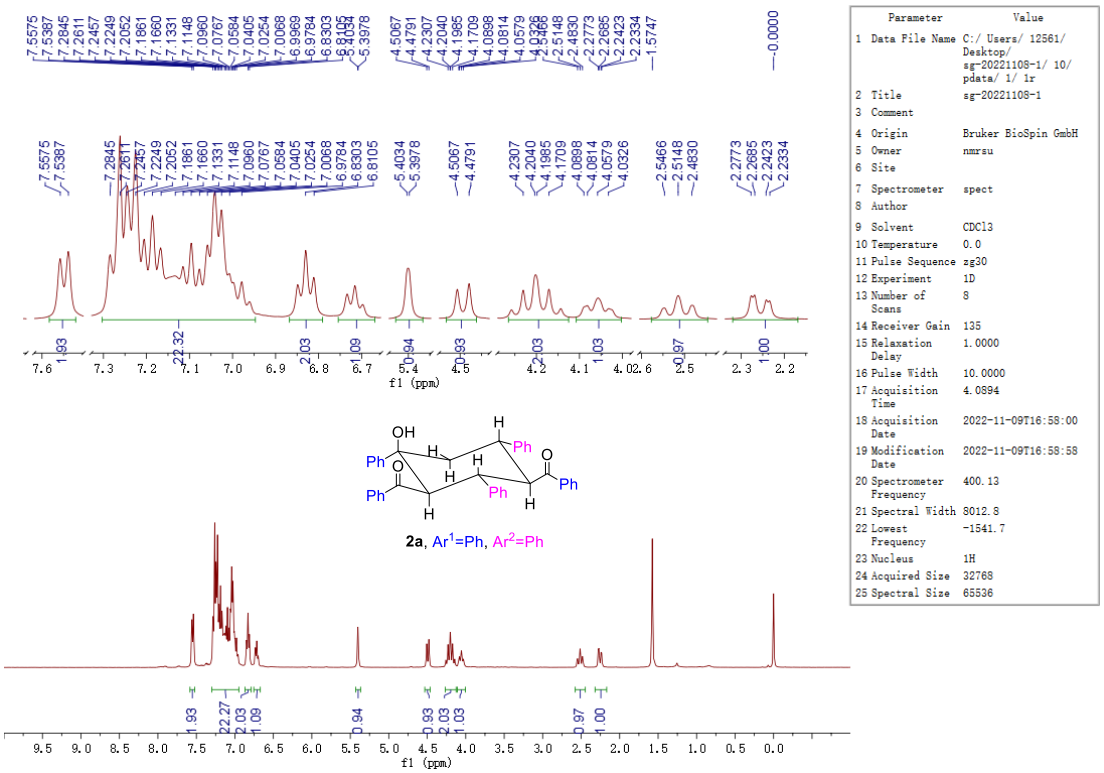
[1] Y. Li, H. Xu, M. Xing, F. Huang, J. Jia and J. Gao, *Org. Lett.*, 2015, **17**, 3690–3693.

[2] (a) Y. Zhang, X. Wu, L. Hao, Z.-R. Wong, S. J. L. Lauw, S. Yang, R. D. Webster and Y. R. Chi, *Org. Chem. Front.*, 2017, **4**, 467–471; (b) J. Zheng, S. Fan, S. Liu, G. Shen, W.-D. Si, X. Dong, X. Huang, Y. Zhang, Q. Yao, Z. Li and D. Sun, *Inorg. Chem. Front.*, 2022, **9**, 5607–5615; (c) H. Gezegen and M. Ceylan, *Synth. Commun.*, 2015, **45**, 2344–2349.

[3] (a) Z. Shan, X. Hu, L. Hu and X. Peng, *Helv. Chim. Acta*, 2009, **92**, 1102–1111.

[4] S. Mukhtar, M. A. Alsharif, M. I. Alahmdi and H. Parveen, *Asian J. Chem.*, 2018, **30**, 1102–1108.

8. NMR spectra of products 2a−2v, 2a′, 3a, 4a and 4q



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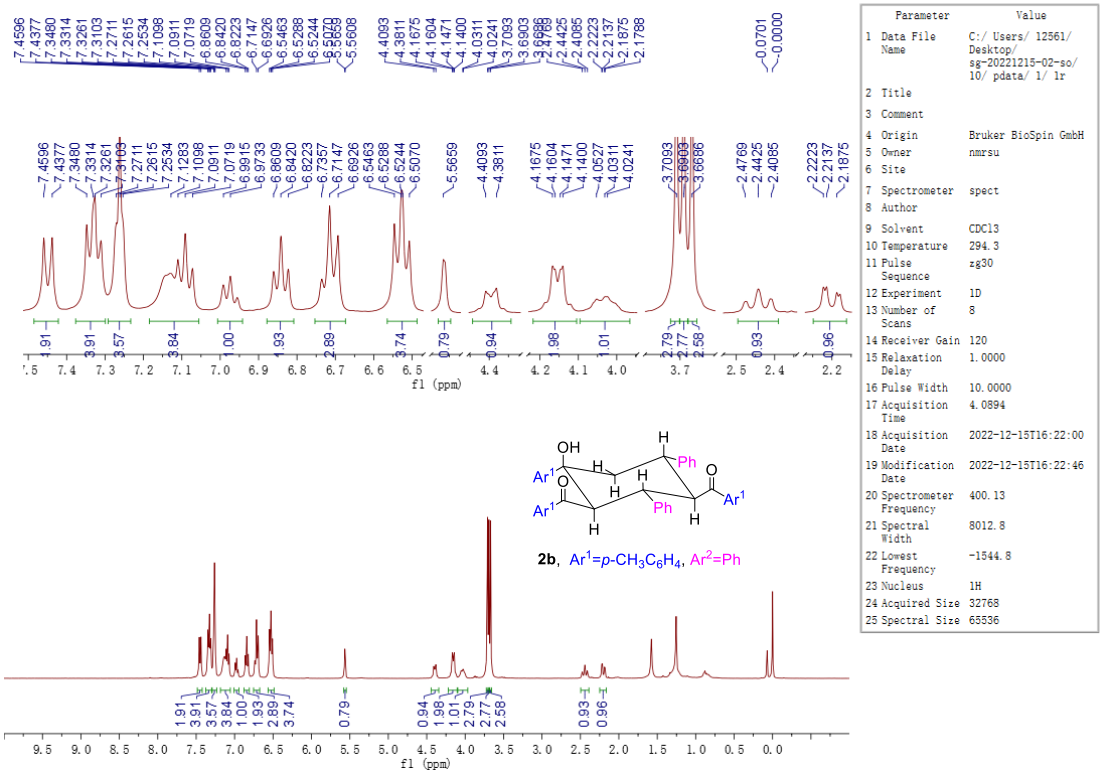
10	Temperature	0.0
11	Pulse Sequence	zg30
12	Experiment	1D
13	Number of Scans	8
14	Receiver Gain	135
15	Relaxation Delay	1.0000
16	Pulse Width	10.0000
17	Acquisition Time	4.0894
18	Acquisition Date	2022-11-09T16:58:00
19	Modification Date	2022-11-09T16:58:58
20	Spectrometer Frequency	400.13
21	Spectral Width	8012.8
22	Lowest Frequency	-1541.7
23	Nucleus	1H
24	Acquired Size	32768
25	Spectral Size	65536

Figure S3 ^{1}H NMR (400 MHz, $CDCl_3$) of compound **2a**



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7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	294.3
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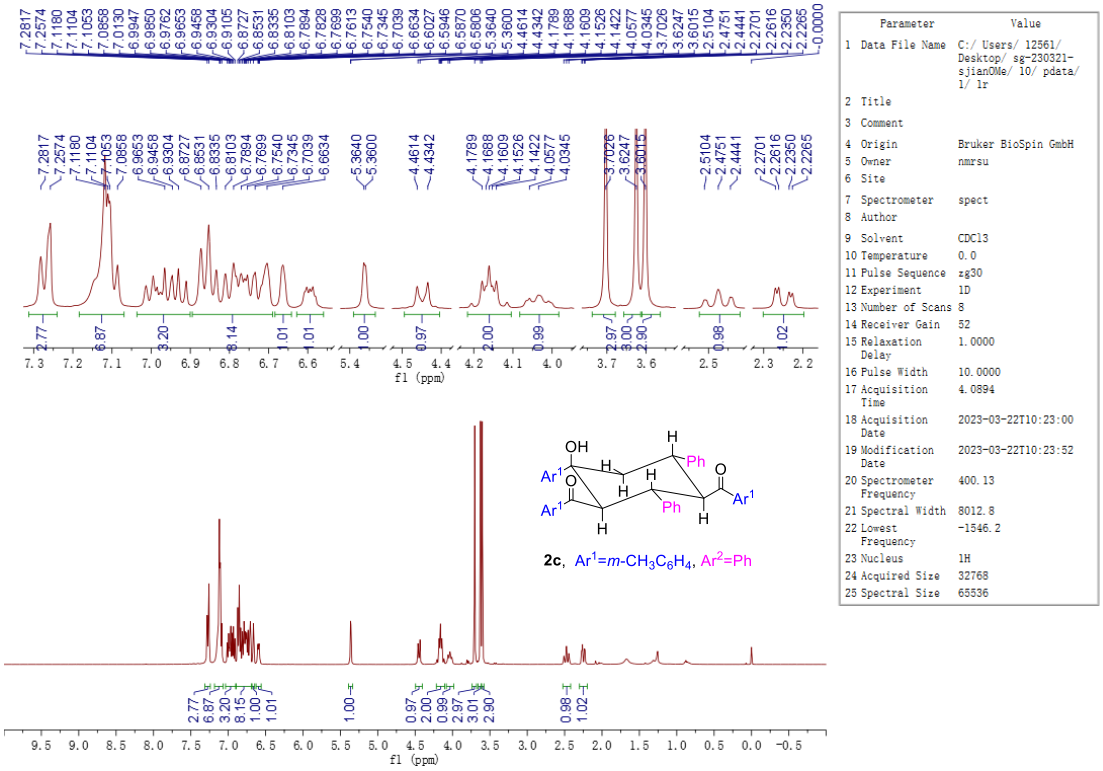
12	Experiment	1D
13	Number of Scans	8
14	Receiver Gain	120
15	Relaxation Delay	1.0000
16	Pulse Width	10.0000
17	Acquisition Time	4.0894
18	Acquisition Date	2022-12-15T16:22:00
19	Modification Date	2022-12-15T16:22:46
20	Spectrometer Frequency	400.13
21	Spectral Width	8012.8
22	Lowest Frequency	-1544.8
23	Nucleus	1H
24	Acquired Size	32768
25	Spectral Size	65536

Figure S4 ^{1}H NMR (400 MHz, $CDCl_3$) of compound **2b**



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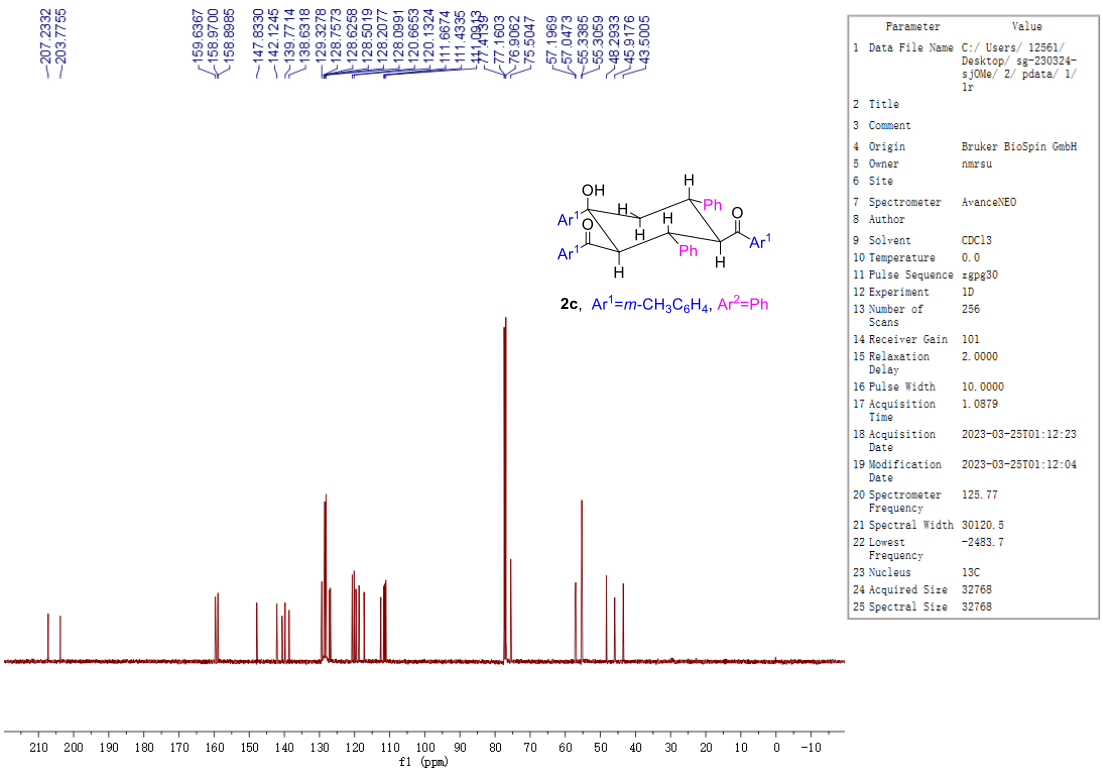
8	Author	
9	Solvent	CDCl3
10	Temperature	0.0
11	Pulse Sequence	zg30
12	Experiment	1D
13	Number of Scans	8
14	Receiver Gain	52
15	Relaxation Delay	1.0000
16	Pulse Width	10.0000
17	Acquisition Time	4.0894
18	Acquisition Date	2023-03-22T10:23:00
19	Modification Date	2023-03-22T10:23:52
20	Spectrometer Frequency	400.13
21	Spectral Width	8012.8
22	Lowest Frequency	-1546.2
23	Nucleus	1H
24	Acquired Size	32768
25	Spectral Size	65536

Figure S5 ^{1}H NMR (400 MHz, $CDCl_3$) of compound **2c**



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7	Spectrometer	AvanceNEO
8	Author	
9	Solvent	CDCl3
10	Temperature	0.0
11	Pulse Sequence	zgpg30
12	Experiment	1D
13	Number of Scans	256
14	Receiver Gain	101
15	Relaxation Delay	2.0000
16	Pulse Width	10.0000
17	Acquisition Time	1.0879
18	Acquisition Date	2023-03-25T01:12:23
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20	Spectrometer Frequency	125.77
21	Spectral Width	30120.5
22	Lowest Frequency	-2483.7
23	Nucleus	13C
24	Acquired Size	32768
25	Spectral Size	32768

Figure S6 ^{13}C NMR (126 MHz, $CDCl_3$) of compound **2c**

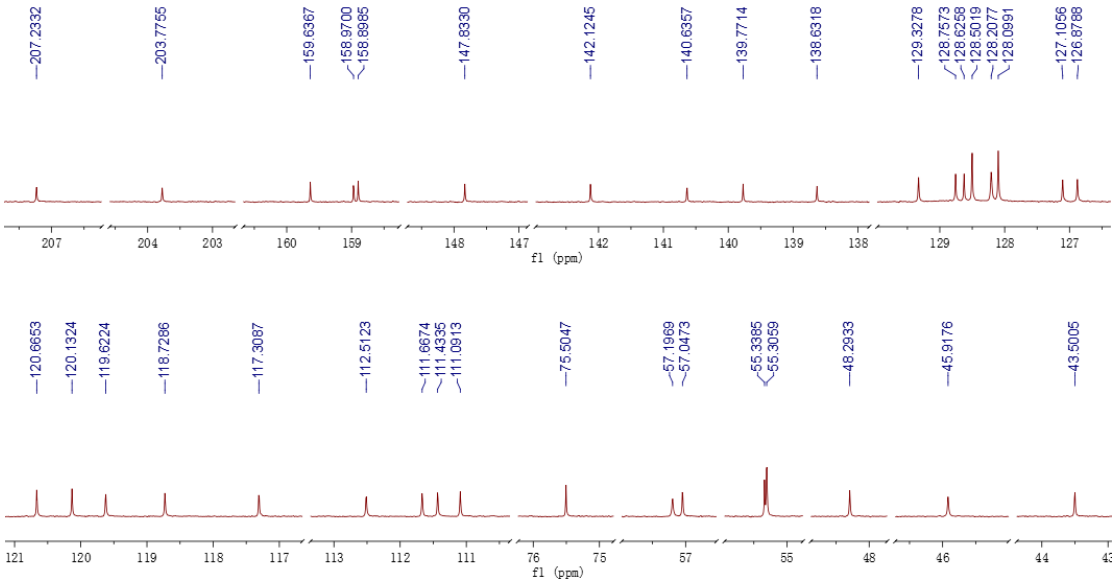


Figure S7 Expanded ^{13}C NMR (126 MHz, $CDCl_3$) of compound **2c**

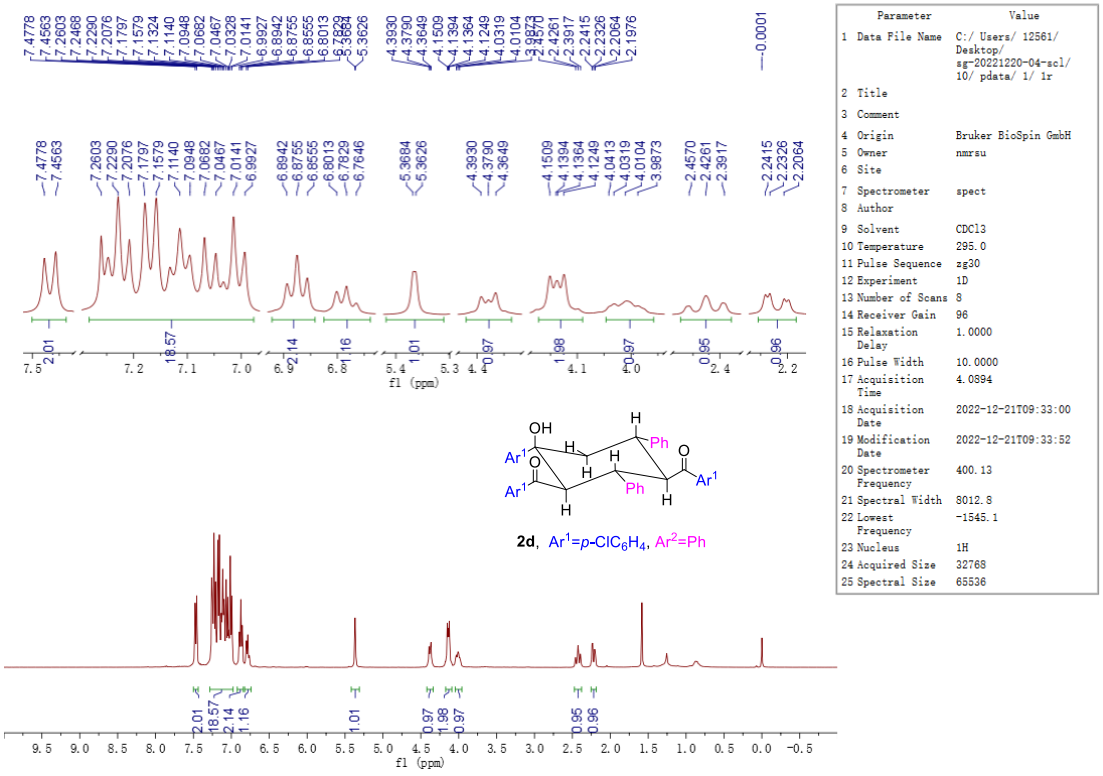
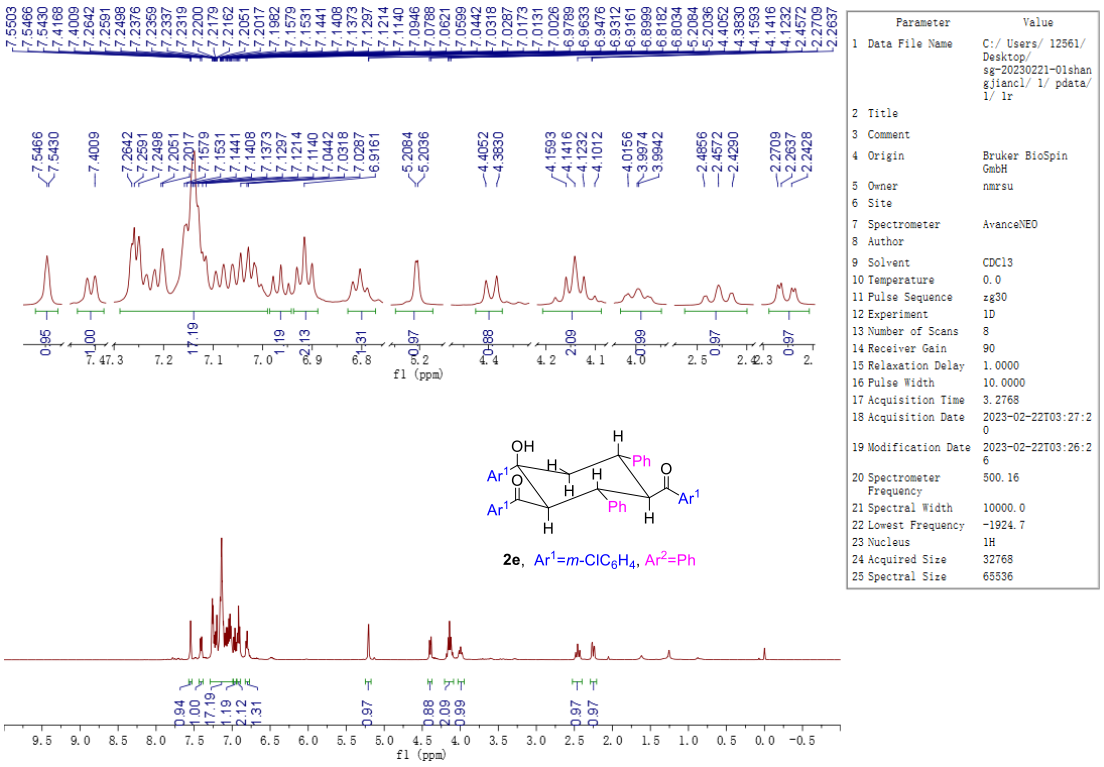


Figure S8 ^{1}H NMR (400 MHz, $CDCl_3$) of compound **2d**



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8	Author	
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10	Temperature	0.0
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12	Experiment	1D
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15	Relaxation Delay	1.0000
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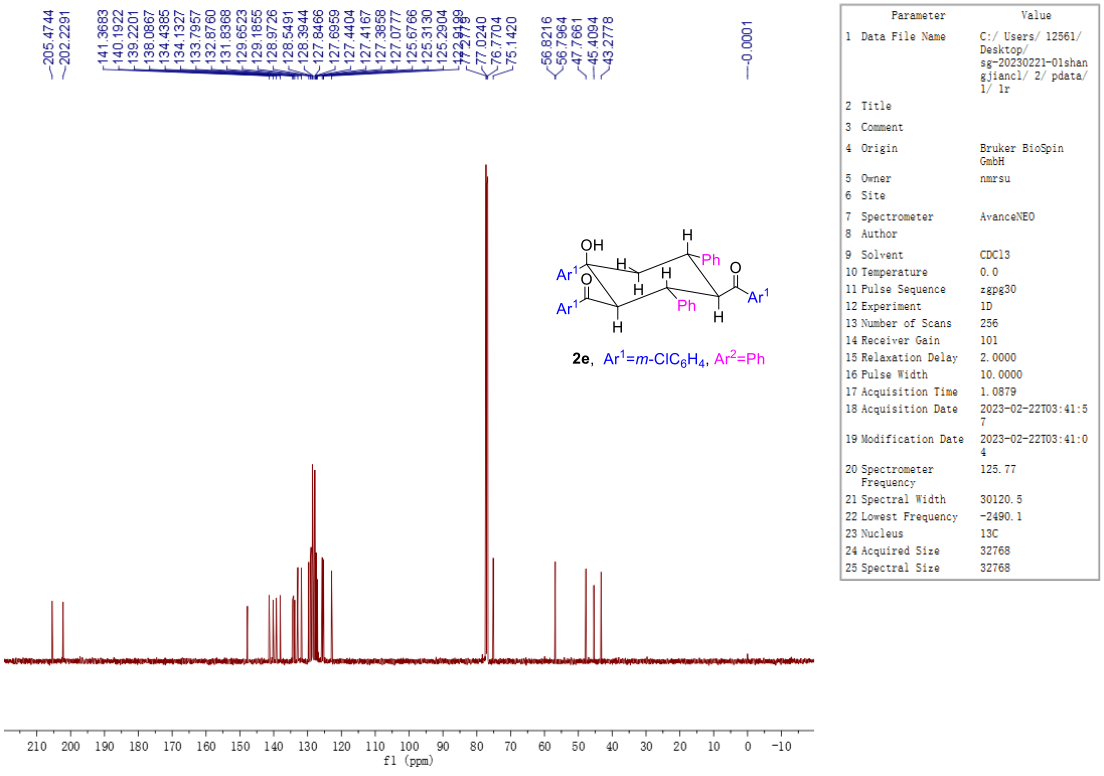
22	Lowest Frequency	-1924.7
23	Nucleus	1H
24	Acquired Size	32768
25	Spectral Size	65536

Figure S9 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **2e**



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8	Author	
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12	Experiment	1D
13	Number of Scans	256
14	Receiver Gain	101
15	Relaxation Delay	2.0000
16	Pulse Width	10.0000
17	Acquisition Time	1.0879
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20	Spectrometer Frequency	125.77
21	Spectral Width	30120.5
22	Lowest Frequency	-2490.1
23	Nucleus	13C
24	Acquired Size	32768
25	Spectral Size	32768

Figure S10 ^{13}C NMR (126 MHz, $CDCl_3$) of compound **2e**

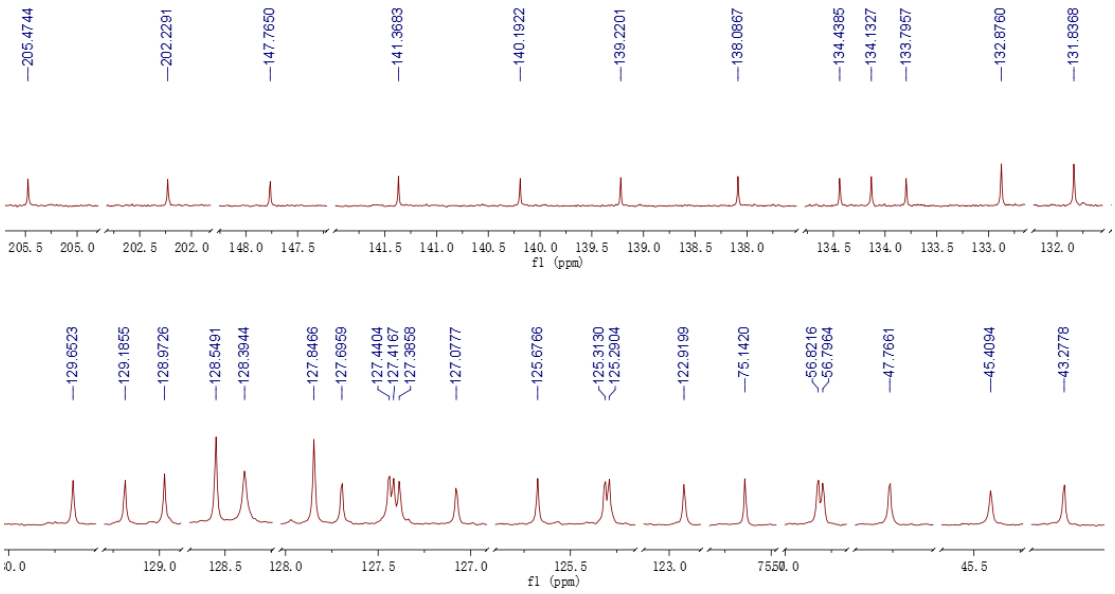


Figure S11 Expanded ^{13}C NMR (126 MHz, $CDCl_3$) of compound **2e**

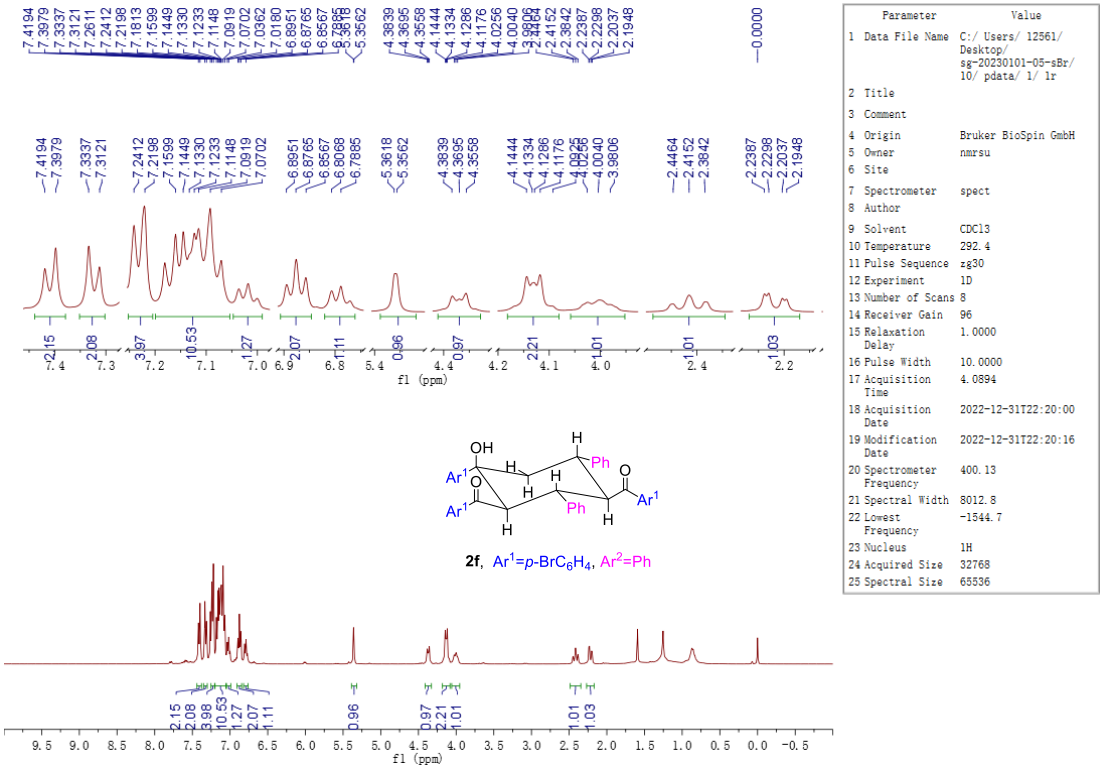
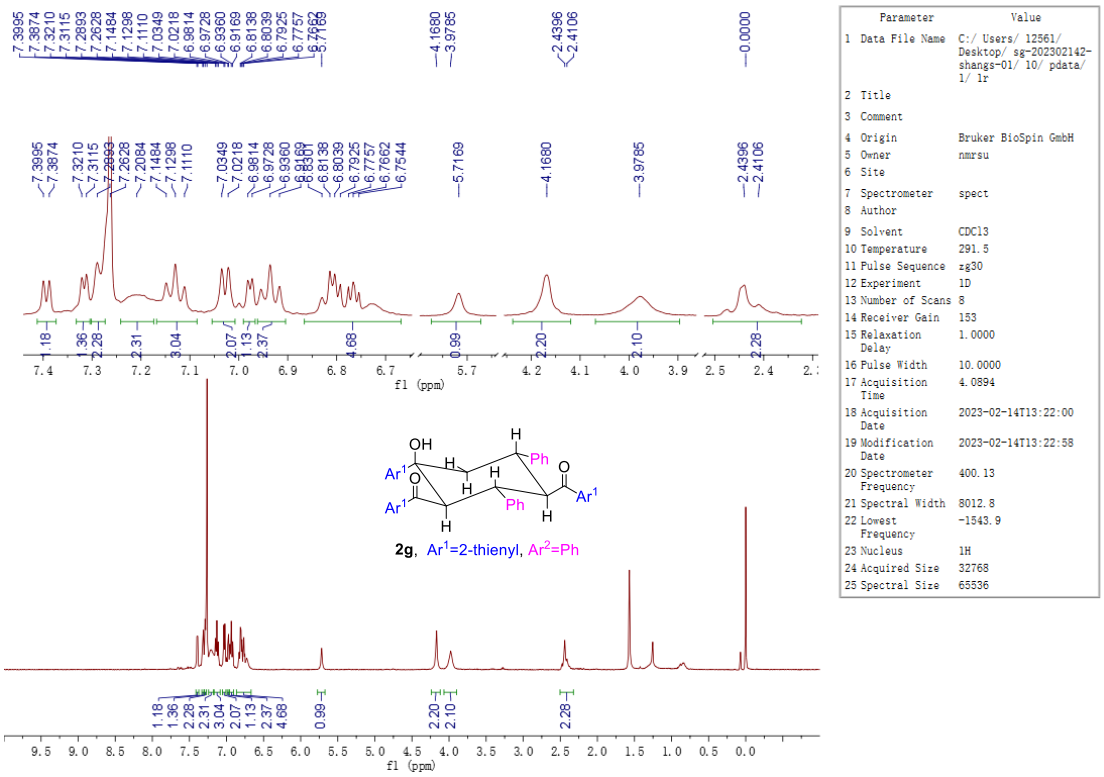


Figure S12 ^1H NMR (400 MHz, $CDCl_3$) of compound **2f**



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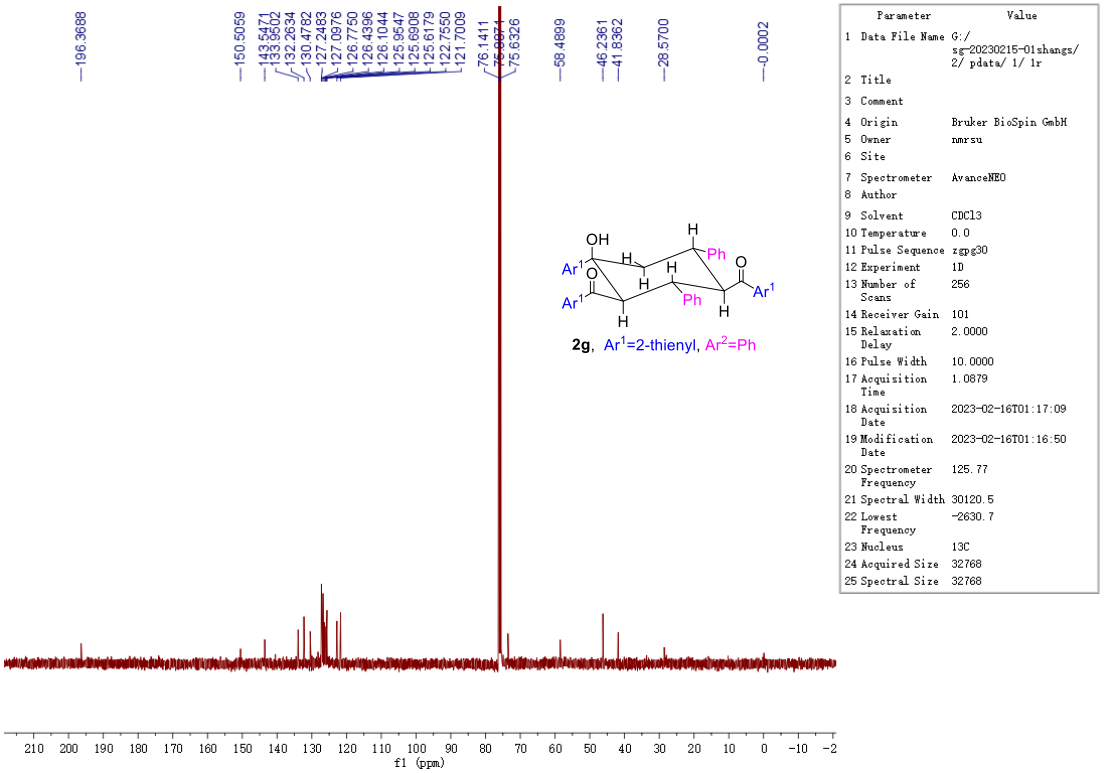
21	Spectral Width	8012.8
22	Lowest Frequency	-1543.9
23	Nucleus	1H
24	Acquired Size	32768
25	Spectral Size	65536

Figure S13 ^{1}H NMR (400 MHz, $CDCl_3$) of compound **2g**



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8	Author	
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12	Experiment	1D
13	Number of Scans	256
14	Receiver Gain	101
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16	Pulse Width	10.0000
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20	Spectrometer Frequency	125.77
21	Spectral Width	30120.5
22	Lowest Frequency	-2630.7
23	Nucleus	13C
24	Acquired Size	32768
25	Spectral Size	32768

Figure S14 ^{13}C NMR (126 MHz, $CDCl_3$) of compound **2g**

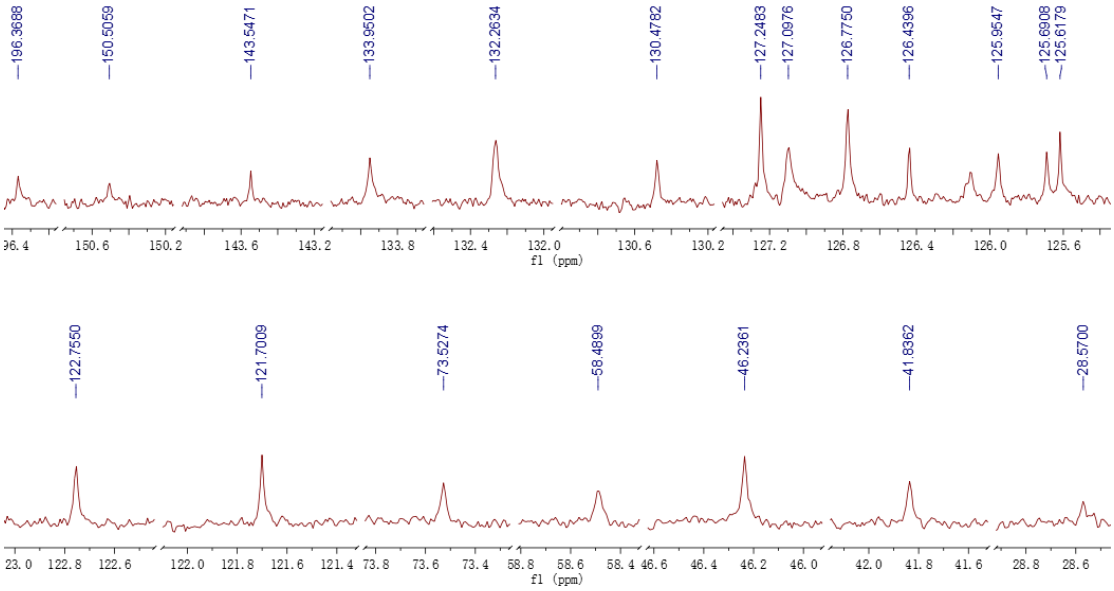


Figure S15 Expanded ^{13}C NMR (126 MHz, $CDCl_3$) of compound **2g**

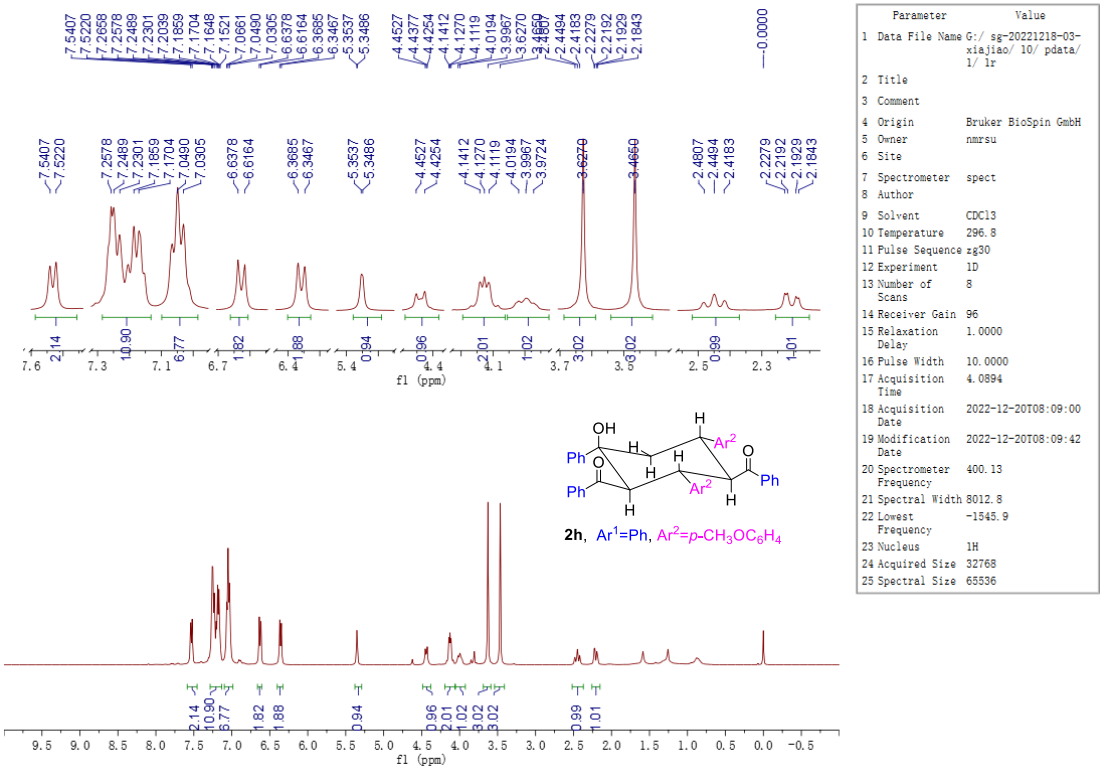
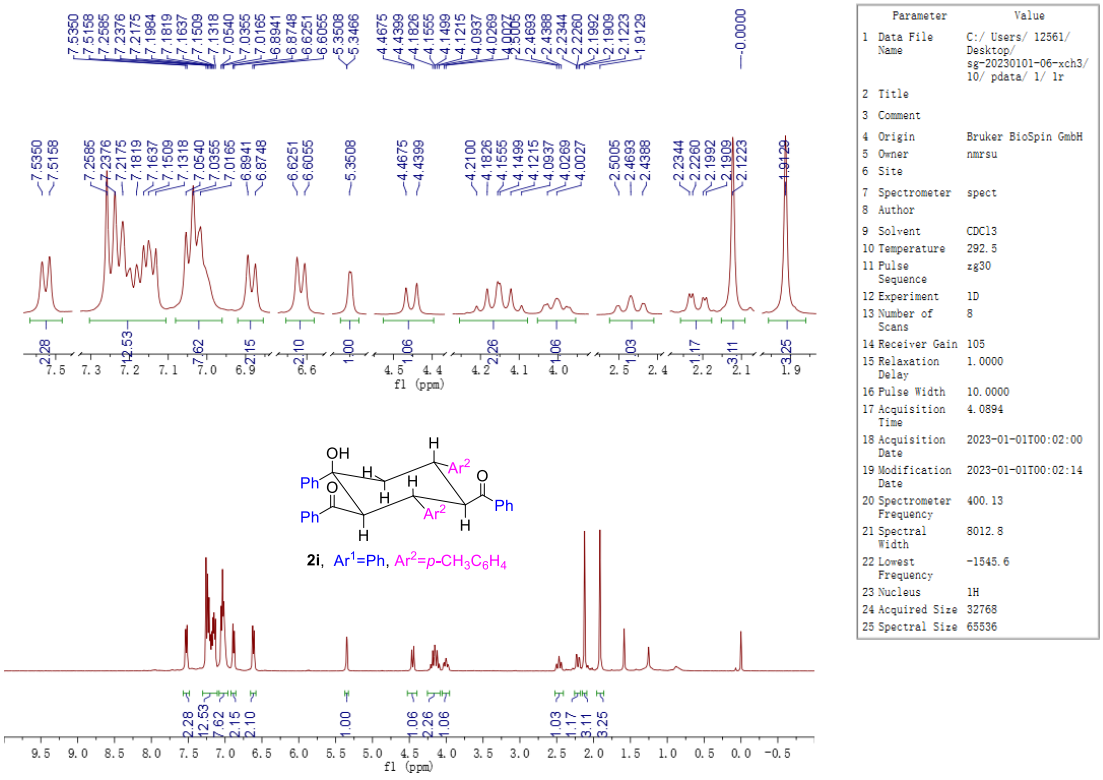


Figure S16 ^{1}H NMR (400 MHz, $CDCl_3$) of compound **2h**



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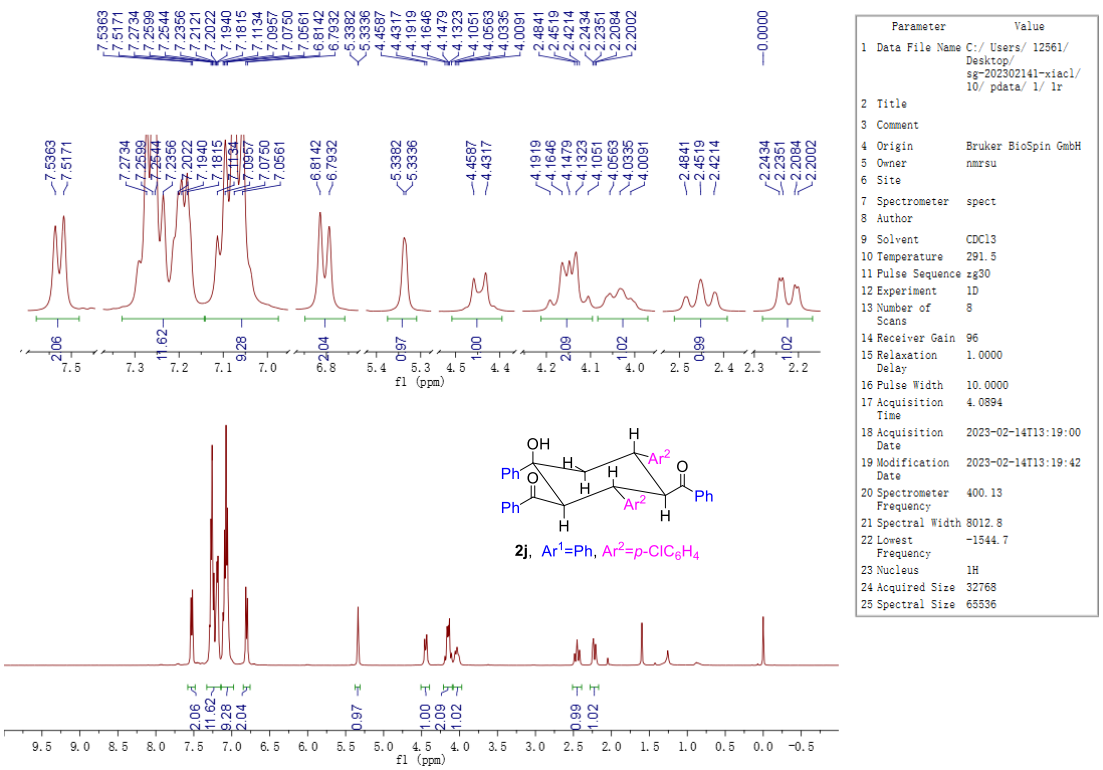
21	Spectral Width	8012.8
22	Lowest Frequency	-1545.6
23	Nucleus	1H
24	Acquired Size	32768
25	Spectral Size	65536

Figure S17 ^{1}H NMR (400 MHz, $CDCl_3$) of compound **2i**



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8	Author	
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11	Pulse Sequence	zg30
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13	Number of Scans	8
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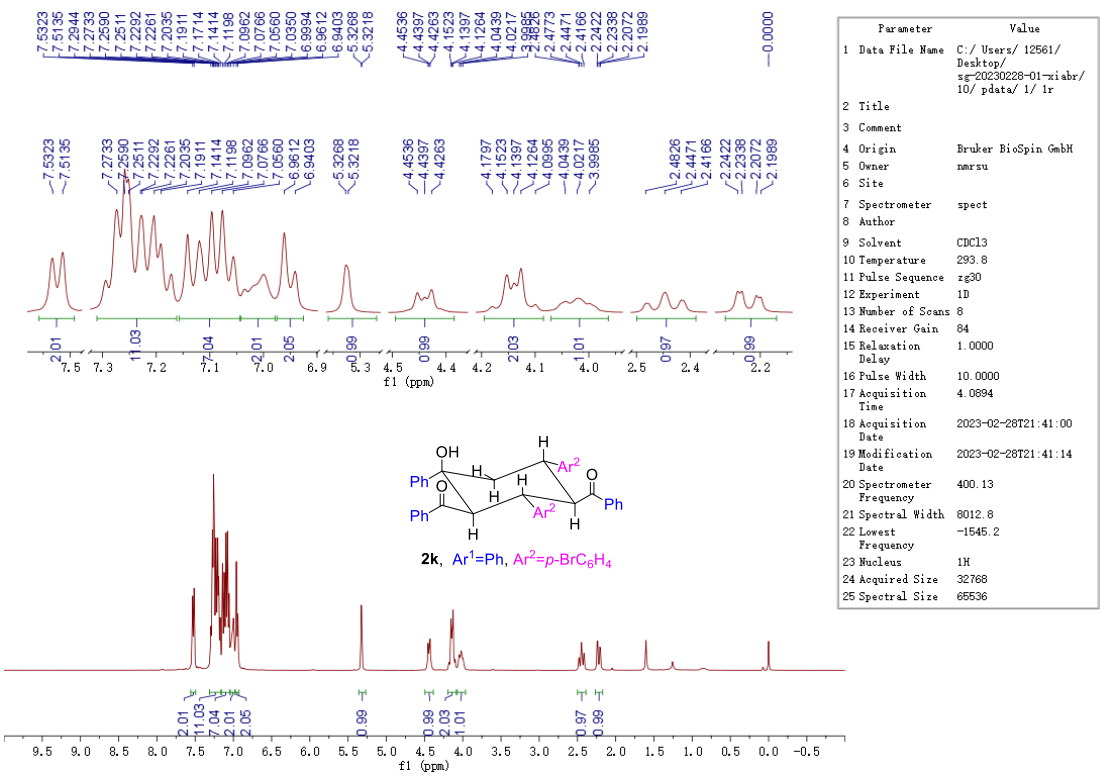
21	Spectral Width	8012.8
22	Lowest Frequency	-1544.7
23	Nucleus	1H
24	Acquired Size	32768
25	Spectral Size	65536

Figure S18 ^{1}H NMR (400 MHz, $CDCl_3$) of compound **2j**



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12	Experiment	1D
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16	Pulse Width	10.0000
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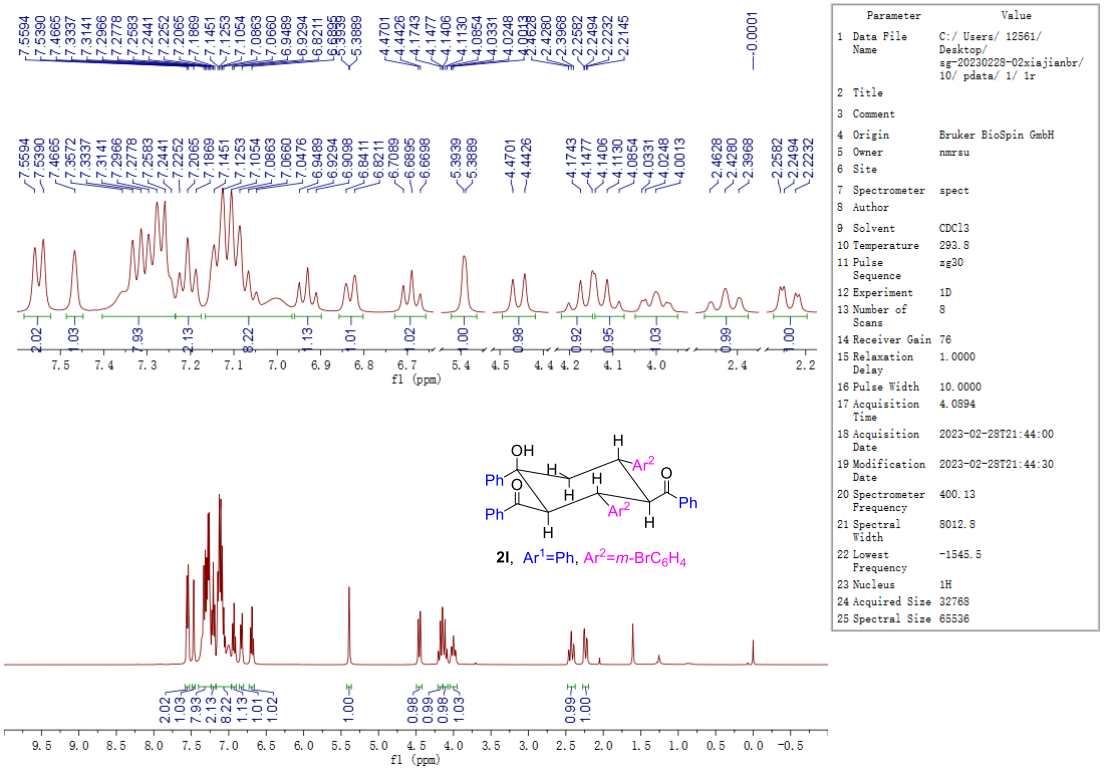
21	Spectral Width	8012.8
22	Lowest Frequency	-1545.2
23	Nucleus	1H
24	Acquired Size	32768
25	Spectral Size	65536

Figure S19 ^{1}H NMR (400 MHz, $CDCl_3$) of compound **2k**



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8	Author	
9	Solvent	CDCl3
10	Temperature	293.8
11	Pulse Sequence	zg30
12	Experiment	1D
13	Number of Scans	8
14	Receiver Gain	76
15	Relaxation Delay	1.0000
16	Pulse Width	10.0000
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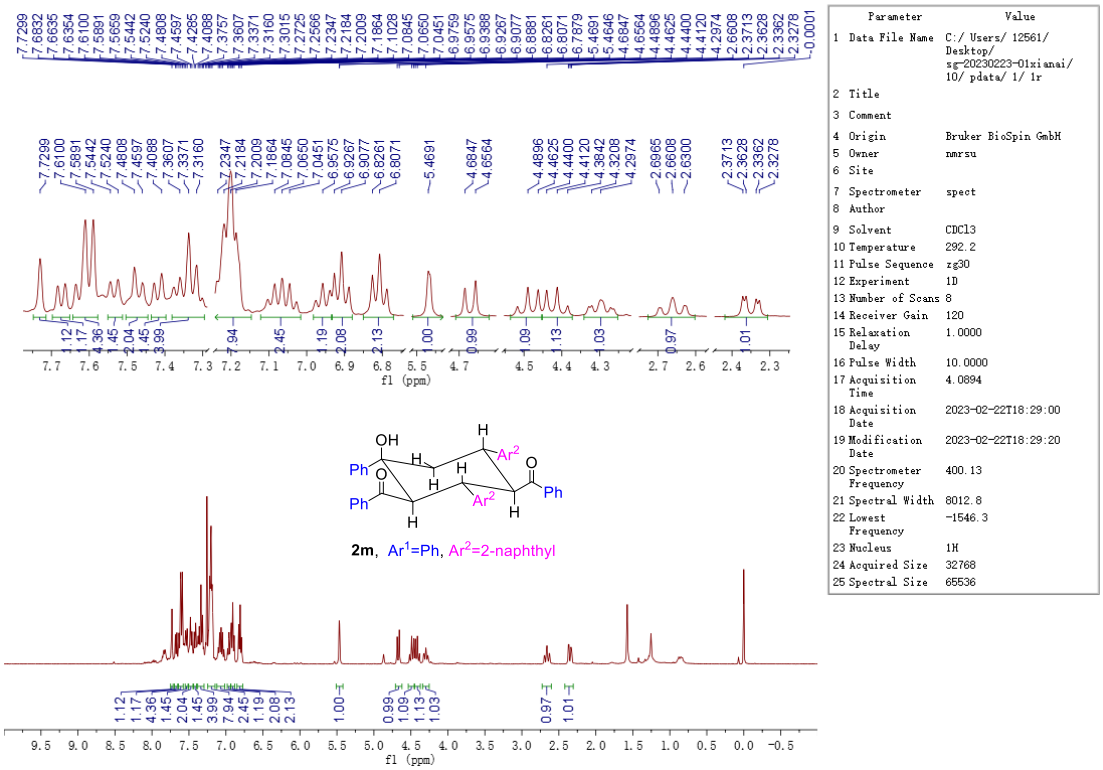
19	Modification Date	2023-02-28T21:44:30
20	Spectrometer Frequency	400.13
21	Spectral Width	8012.8
22	Lowest Frequency	-1545.5
23	Nucleus	1H
24	Acquired Size	32768
25	Spectral Size	65536

Figure S20 ^{1}H NMR (400 MHz, $CDCl_3$) of compound **2l**



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5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	292.2
11	Pulse Sequence	zg30
12	Experiment	1D
13	Number of Scans	8
14	Receiver Gain	120
15	Relaxation Delay	1.0000
16	Pulse Width	10.0000
17	Acquisition Time	4.0894
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19	Modification Date	2023-02-22T18:29:20
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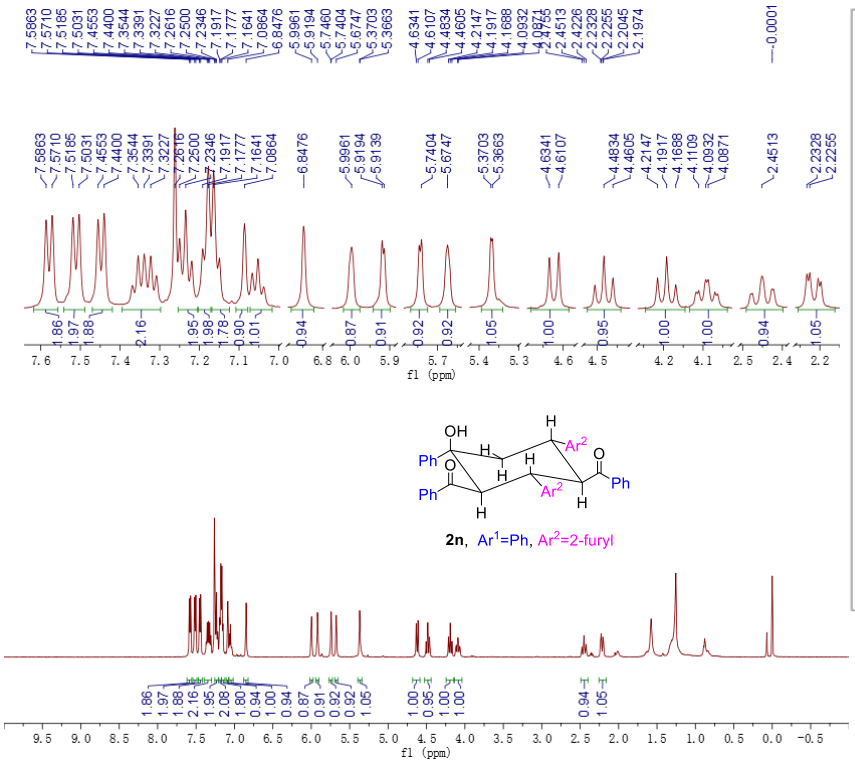
21	Spectral Width	8012.8
22	Lowest Frequency	-1546.3
23	Nucleus	1H
24	Acquired Size	32768
25	Spectral Size	65536

Figure S21 ^{1}H NMR (400 MHz, $CDCl_3$) of compound **2m**



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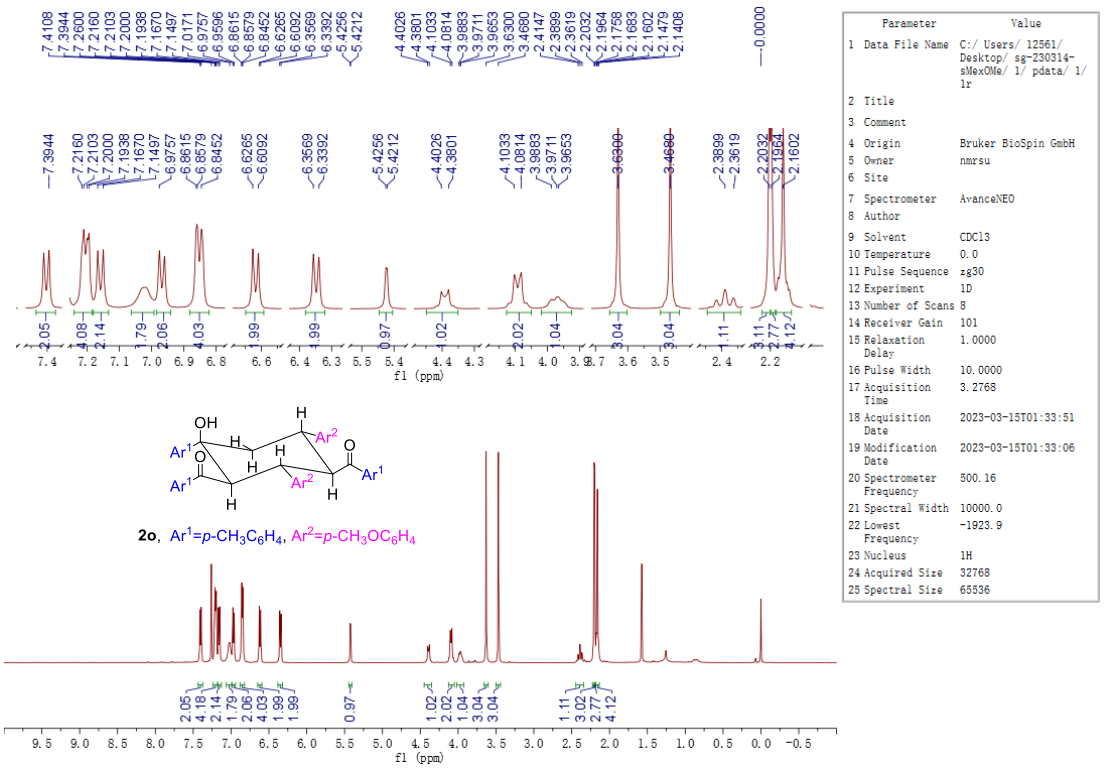
5	Owner	nmrsu
6	Site	
7	Spectrometer	AvanceNEO
8	Author	
9	Solvent	CDCl3
10	Temperature	0.0
11	Pulse Sequence	zg30
12	Experiment	1D
13	Number of Scans	8
14	Receiver Gain	101
15	Relaxation Delay	1.0000
16	Pulse Width	10.0000
17	Acquisition Time	3.2768
18	Acquisition Date	2023-03-08T02:21:38
19	Modification Date	2023-03-08T02:21:34
20	Spectrometer Frequency	500.16
21	Spectral Width	10000.0
22	Lowest Frequency	-1923.4
23	Nucleus	1H
24	Acquired Size	32768
25	Spectral Size	65536

Figure S22 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **2n**



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5	Owner	nmrsu
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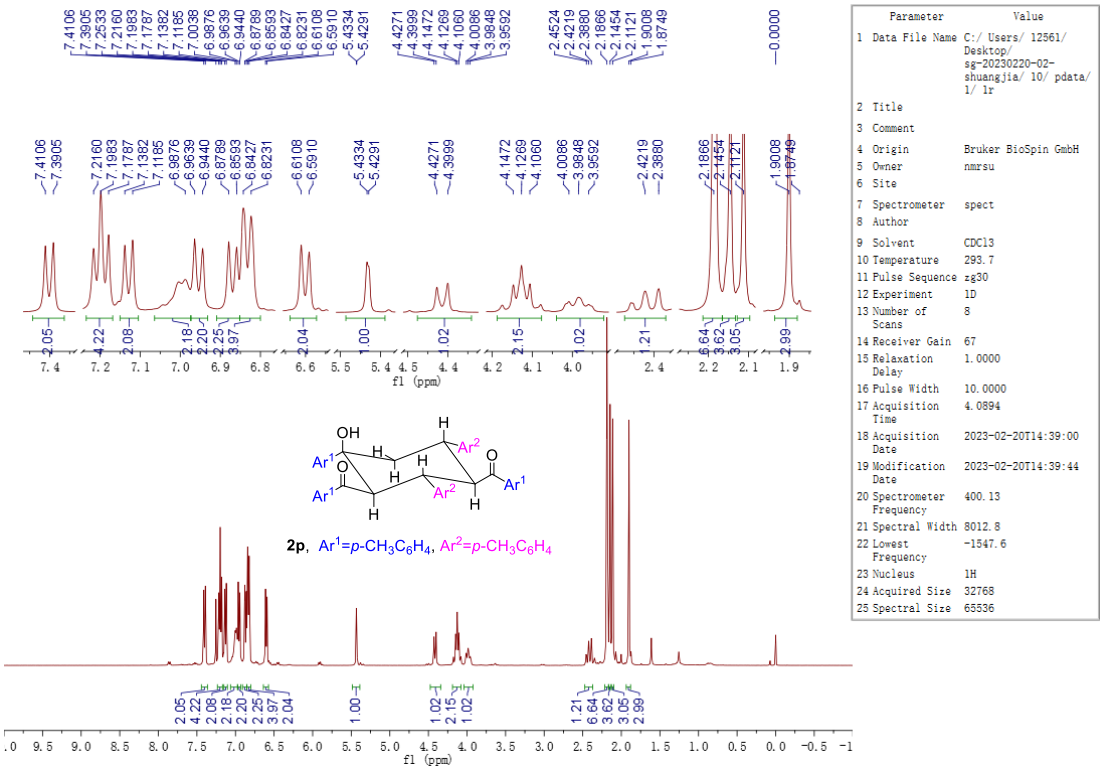
7	Spectrometer	AvanceNEO
8	Author	
9	Solvent	CDCl3
10	Temperature	0.0
11	Pulse Sequence	zg30
12	Experiment	1D
13	Number of Scans	8
14	Receiver Gain	101
15	Relaxation Delay	1.0000
16	Pulse Width	10.0000
17	Acquisition Time	3.2768
18	Acquisition Date	2023-03-15T01:33:51
19	Modification Date	2023-03-15T01:33:06
20	Spectrometer Frequency	500.16
21	Spectral Width	10000.0
22	Lowest Frequency	-1923.9
23	Nucleus	1H
24	Acquired Size	32768
25	Spectral Size	65536

Figure S23 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **2o**



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3	Comment	
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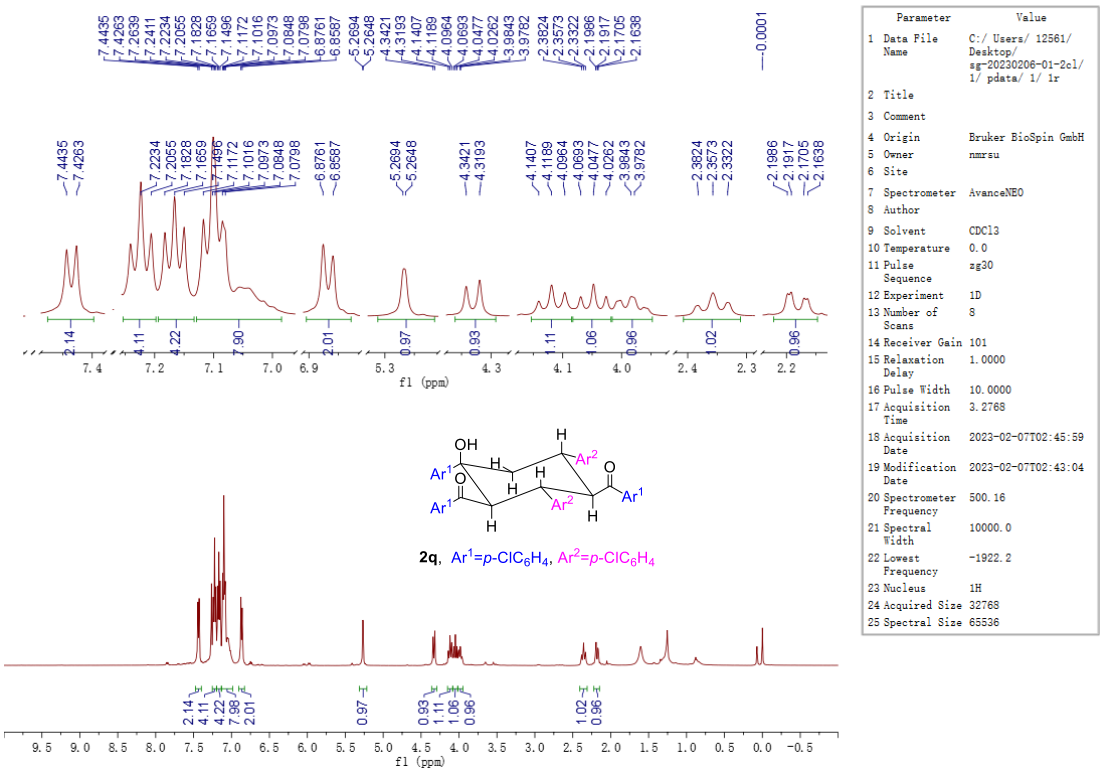
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	293.7
11	Pulse Sequence	zg30
12	Experiment	1D
13	Number of Scans	8
14	Receiver Gain	67
15	Relaxation Delay	1.0000
16	Pulse Width	10.0000
17	Acquisition Time	4.0894
18	Acquisition Date	2023-02-20T14:39:00
19	Modification Date	2023-02-20T14:39:44
20	Spectrometer Frequency	400.13
21	Spectral Width	8012.8
22	Lowest Frequency	-1547.6
23	Nucleus	1H
24	Acquired Size	32768
25	Spectral Size	65536

Figure S24 ^{1}H NMR (400 MHz, $CDCl_3$) of compound **2p**



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3	Comment	
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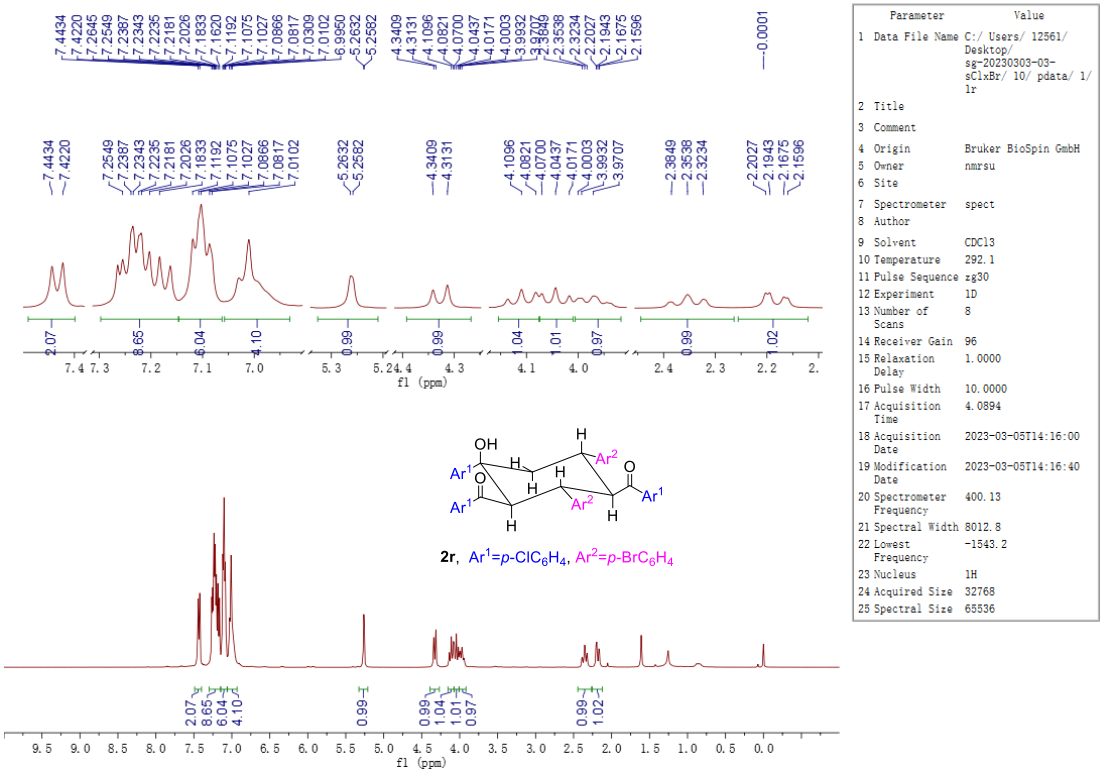
5	Owner	nmrsu
6	Site	
7	Spectrometer	AvanceNEO
8	Author	
9	Solvent	CDCl3
10	Temperature	0.0
11	Pulse Sequence	zg30
12	Experiment	1D
13	Number of Scans	8
14	Receiver Gain	101
15	Relaxation Delay	1.0000
16	Pulse Width	10.0000
17	Acquisition Time	3.2768
18	Acquisition Date	2023-02-07T02:45:59
19	Modification Date	2023-02-07T02:43:04
20	Spectrometer Frequency	500.16
21	Spectral Width	10000.0
22	Lowest Frequency	-1922.2
23	Nucleus	1H
24	Acquired Size	32768
25	Spectral Size	65536

Figure S25 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **2q**



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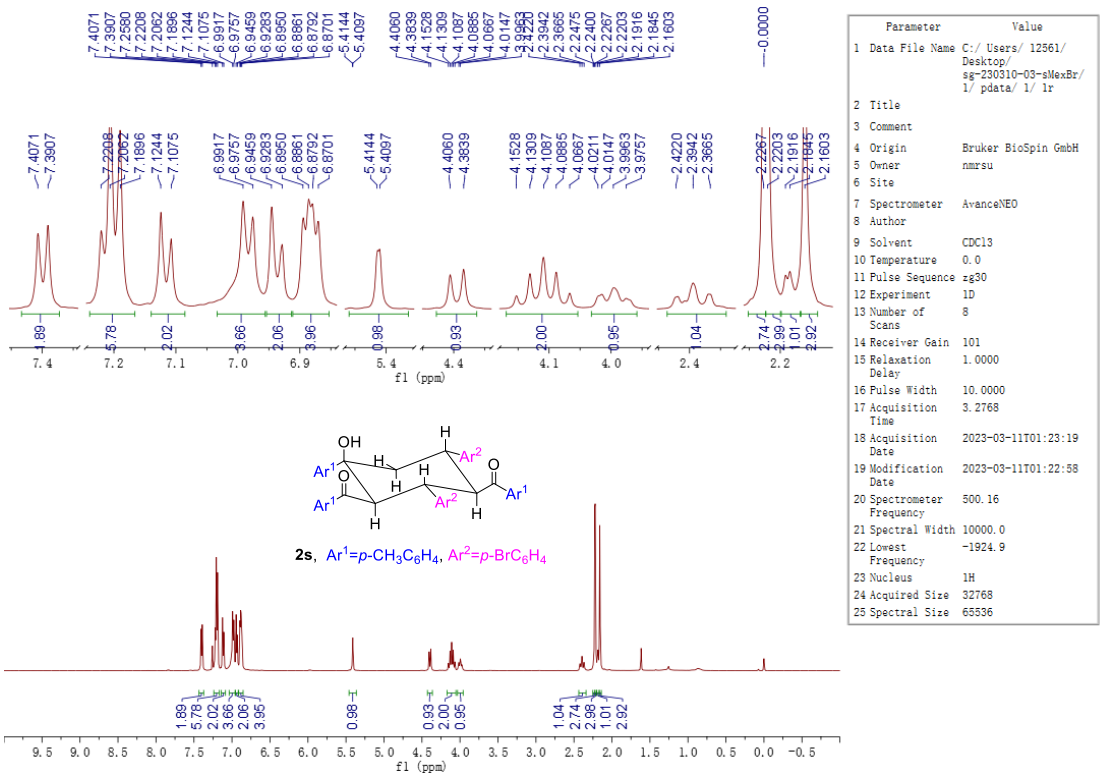
2	Title	
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	292.1
11	Pulse Sequence	zg30
12	Experiment	1D
13	Number of Scans	8
14	Receiver Gain	96
15	Relaxation Delay	1.0000
16	Pulse Width	10.0000
17	Acquisition Time	4.0894
18	Acquisition Date	2023-03-05T14:16:00
19	Modification Date	2023-03-05T14:16:40
20	Spectrometer Frequency	400.13
21	Spectral Width	8012.8
22	Lowest Frequency	-1543.2
23	Nucleus	1H
24	Acquired Size	32768
25	Spectral Size	65536

Figure S26 ^{1}H NMR (400 MHz, $CDCl_3$) of compound **2r**



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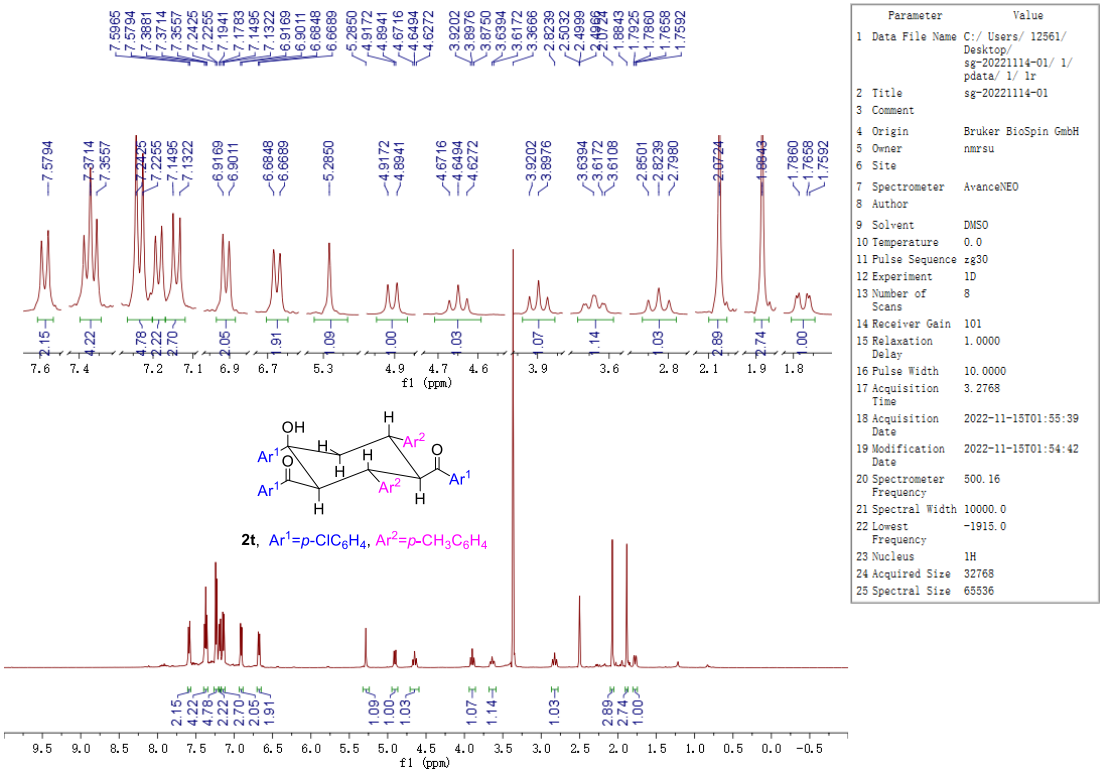
5	Owner	nmrsu
6	Site	
7	Spectrometer	AvanceNEO
8	Author	
9	Solvent	CDCl3
10	Temperature	0.0
11	Pulse Sequence	zg30
12	Experiment	1D
13	Number of Scans	8
14	Receiver Gain	101
15	Relaxation Delay	1.0000
16	Pulse Width	10.0000
17	Acquisition Time	3.2768
18	Acquisition Date	2023-03-11T01:23:19
19	Modification Date	2023-03-11T01:22:58
20	Spectrometer Frequency	500.16
21	Spectral Width	10000.0
22	Lowest Frequency	-1924.9
23	Nucleus	1H
24	Acquired Size	32768
25	Spectral Size	65536

Figure S27 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **2s**



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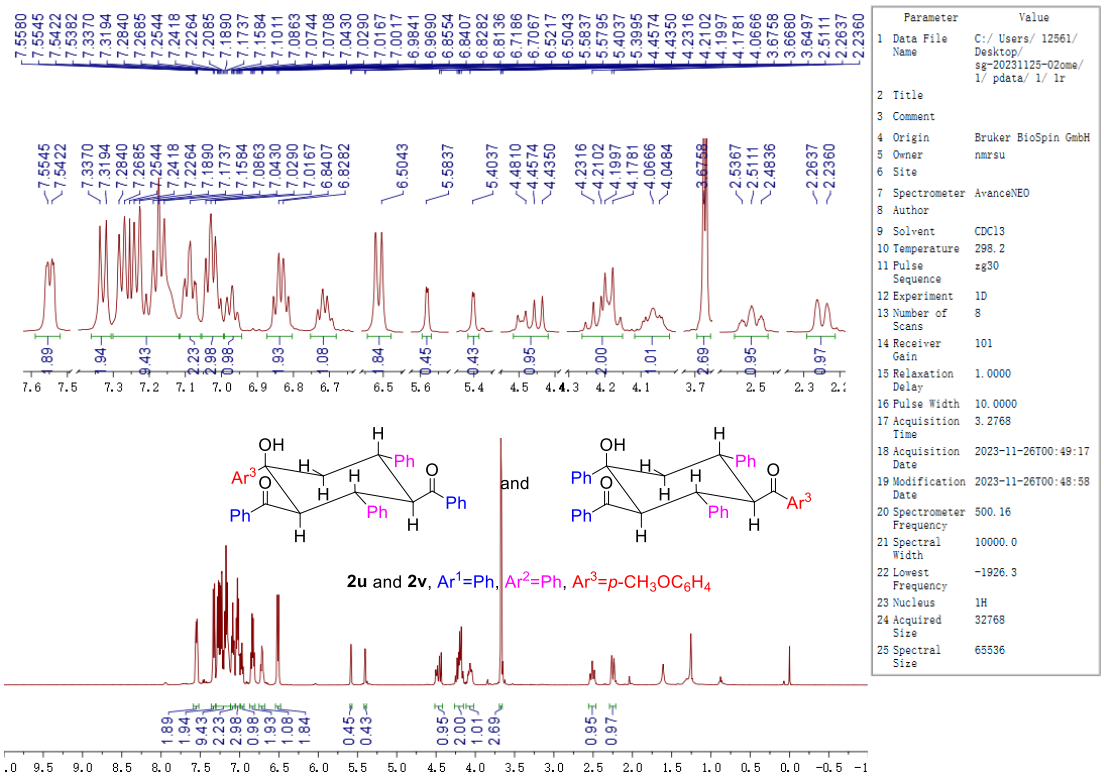
5	Owner	nmrsu
6	Site	
7	Spectrometer	AvanceNEO
8	Author	
9	Solvent	DMSO
10	Temperature	0.0
11	Pulse Sequence	zg30
12	Experiment	1D
13	Number of Scans	8
14	Receiver Gain	101
15	Relaxation Delay	1.0000
16	Pulse Width	10.0000
17	Acquisition Time	3.2768
18	Acquisition Date	2022-11-15T01:55:39
19	Modification Date	2022-11-15T01:54:42
20	Spectrometer Frequency	500.16
21	Spectral Width	10000.0
22	Lowest Frequency	-1915.0
23	Nucleus	1H
24	Acquired Size	32768
25	Spectral Size	65536

Figure S28 ^{1}H NMR (500 MHz, DMSO-d_6) of compound **2t**



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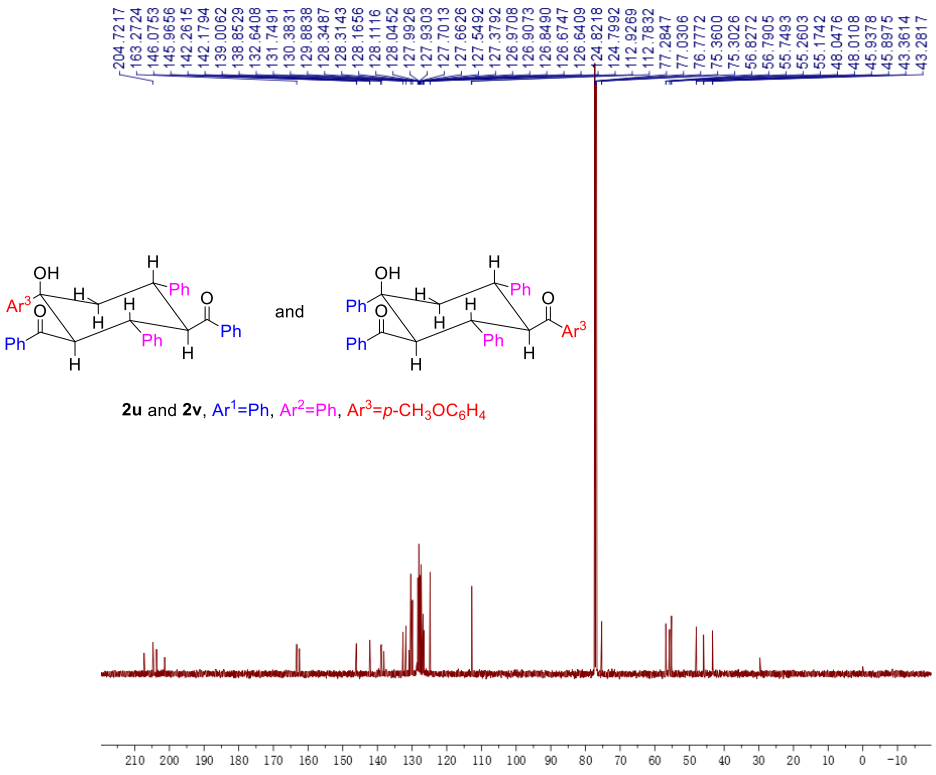
5	Owner	nmrsu
6	Site	
7	Spectrometer	AvanceNEO
8	Author	
9	Solvent	CDCl3
10	Temperature	298.2
11	Pulse Sequence	zg30
12	Experiment	1D
13	Number of Scans	8
14	Receiver Gain	101
15	Relaxation Delay	1.0000
16	Pulse Width	10.0000
17	Acquisition Time	3.2768
18	Acquisition Date	2023-11-26T00:49:17
19	Modification Date	2023-11-26T00:48:58
20	Spectrometer Frequency	500.16
21	Spectral Width	10000.0
22	Lowest Frequency	-1926.3
23	Nucleus	1H
24	Acquired Size	32768
25	Spectral Size	65536

Figure S29 ^{1}H NMR (500 MHz, $CDCl_3$) of compounds **2u** and **2v**



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6	Site	
7	Spectrometer	AvanceNEO
8	Author	
9	Solvent	CDCl3
10	Temperature	298.2
11	Pulse Sequence	zgpg30
12	Experiment	1D
13	Number of Scans	256
14	Receiver Gain	101
15	Relaxation Delay	2.0000
16	Pulse Width	10.0000
17	Acquisition Time	1.0879
18	Acquisition Date	2023-11-26T01:03:35
19	Modification Date	2023-11-26T01:03:16
20	Spectrometer Frequency	125.77
21	Spectral Width	30120.5
22	Lowest Frequency	-2486.5
23	Nucleus	13C
24	Acquired Size	32768
25	Spectral Size	32768

Figure S30 ^{13}C NMR (126 MHz, $CDCl_3$) of compounds **2u** and **2v**

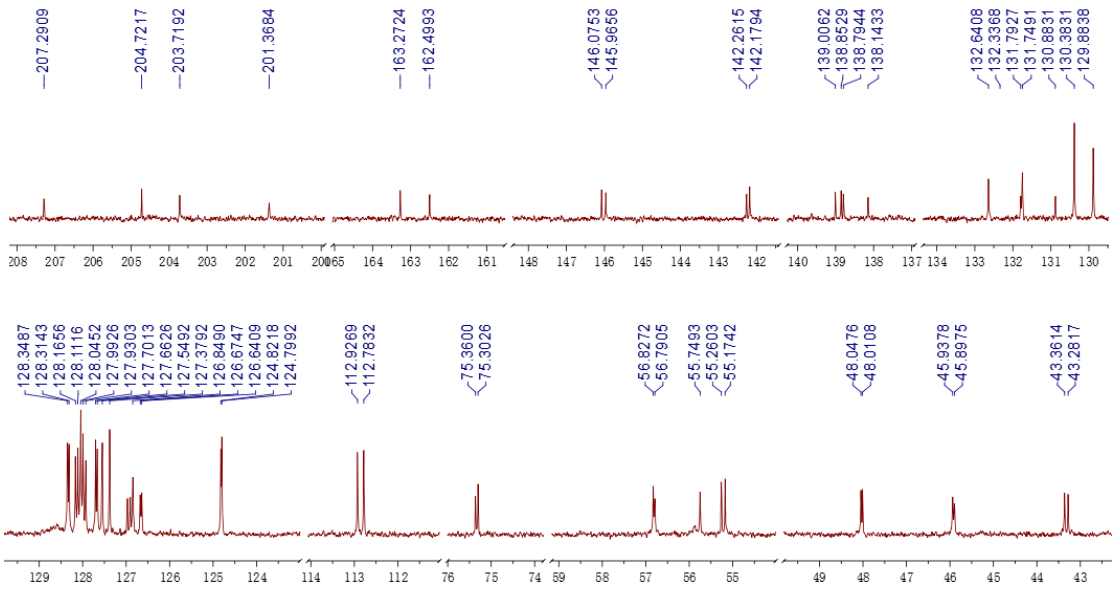
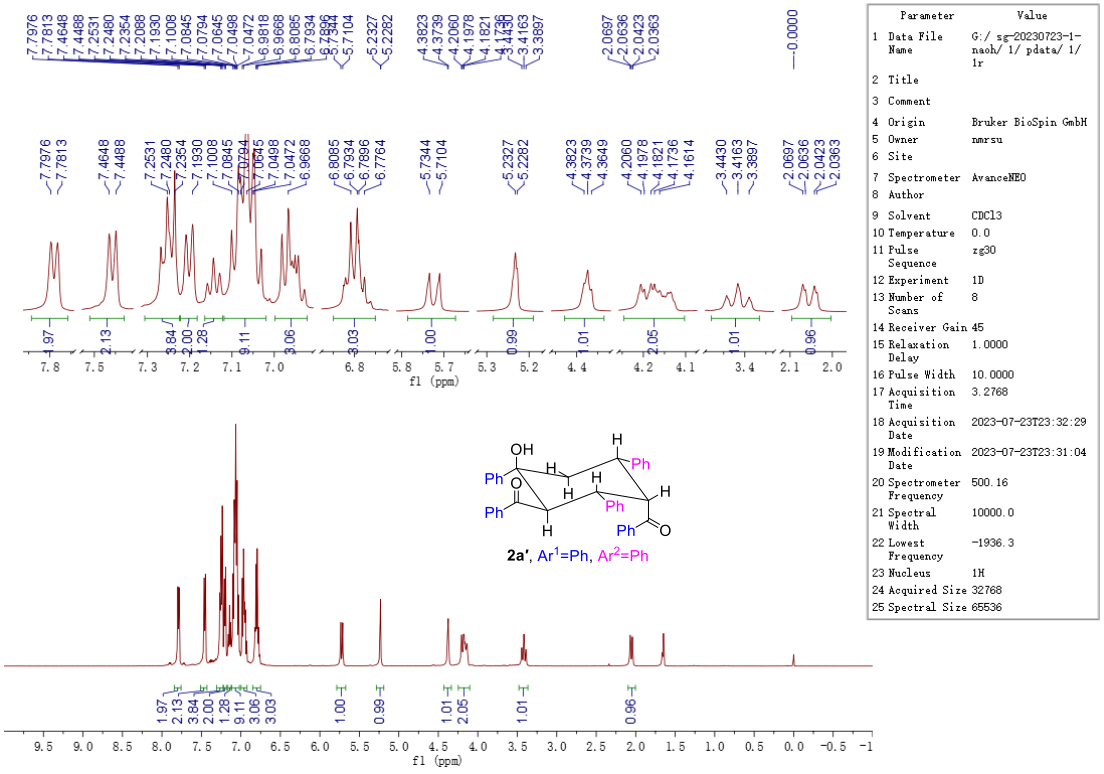


Figure S31 Expanded ^{13}C NMR (126 MHz, $CDCl_3$) of compounds **2u** and **2v**



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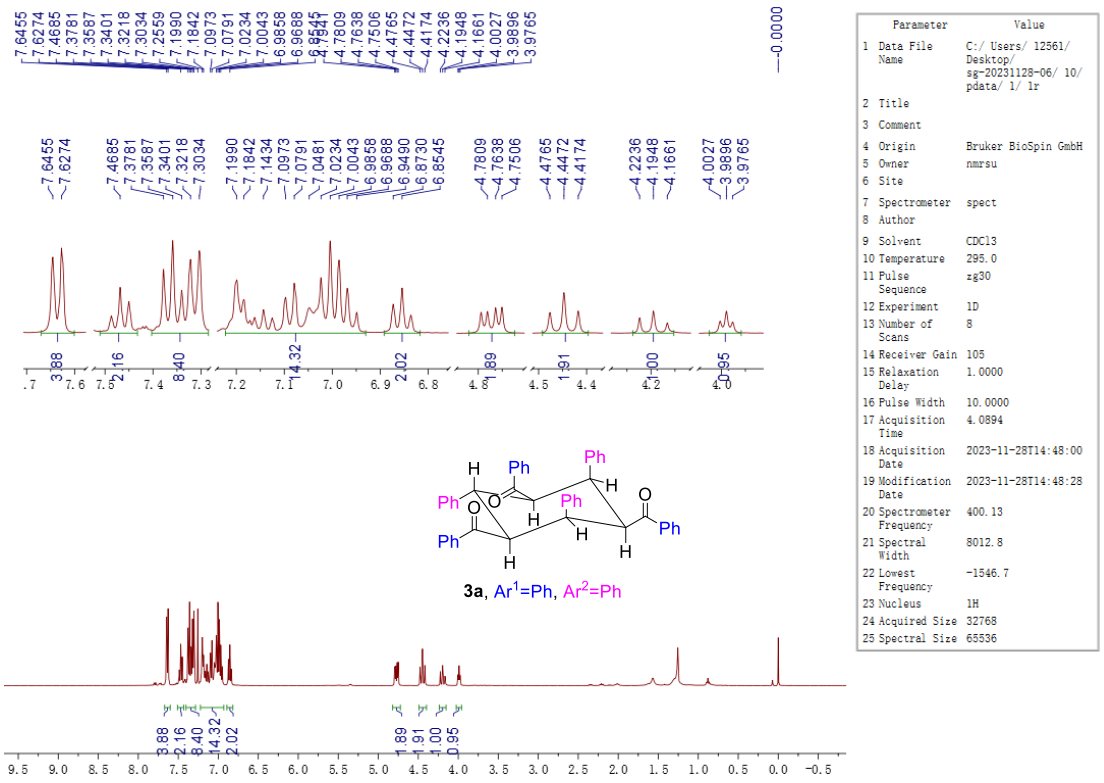
7	Spectrometer	AvanceNEO
8	Author	
9	Solvent	CDCl3
10	Temperature	0.0
11	Pulse Sequence	zg30
12	Experiment	1D
13	Number of Scans	8
14	Receiver Gain	45
15	Relaxation Delay	1.0000
16	Pulse Width	10.0000
17	Acquisition Time	3.2768
18	Acquisition Date	2023-07-23T23:32:29
19	Modification Date	2023-07-23T23:31:04
20	Spectrometer Frequency	500.16
21	Spectral Width	10000.0
22	Lowest Frequency	-1936.3
23	Nucleus	1H
24	Acquired Size	32768
25	Spectral Size	65536

Figure S32 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **2a′**



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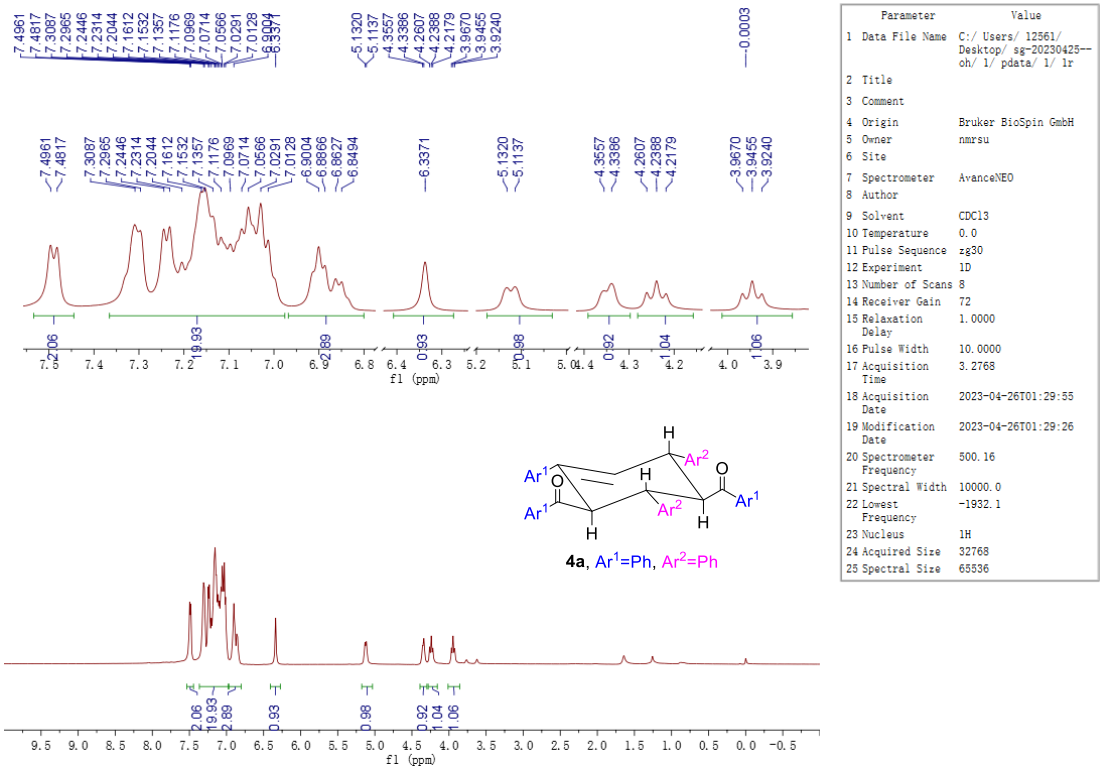
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	CDCl3
10	Temperature	295.0
11	Pulse Sequence	zg30
12	Experiment	1D
13	Number of Scans	8
14	Receiver Gain	105
15	Relaxation Delay	1.0000
16	Pulse Width	10.0000
17	Acquisition Time	4.0894
18	Acquisition Date	2023-11-28T14:48:00
19	Modification Date	2023-11-28T14:48:28
20	Spectrometer Frequency	400.13
21	Spectral Width	8012.8
22	Lowest Frequency	-1546.7
23	Nucleus	1H
24	Acquired Size	32768
25	Spectral Size	65536

Figure S33 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **3a**



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5	Owner	nmrsu
6	Site	
7	Spectrometer	AvanceNEO
8	Author	
9	Solvent	CDCl3
10	Temperature	0.0
11	Pulse Sequence	zg30
12	Experiment	1D
13	Number of Scans	8
14	Receiver Gain	72
15	Relaxation Delay	1.0000
16	Pulse Width	10.0000
17	Acquisition Time	3.2768
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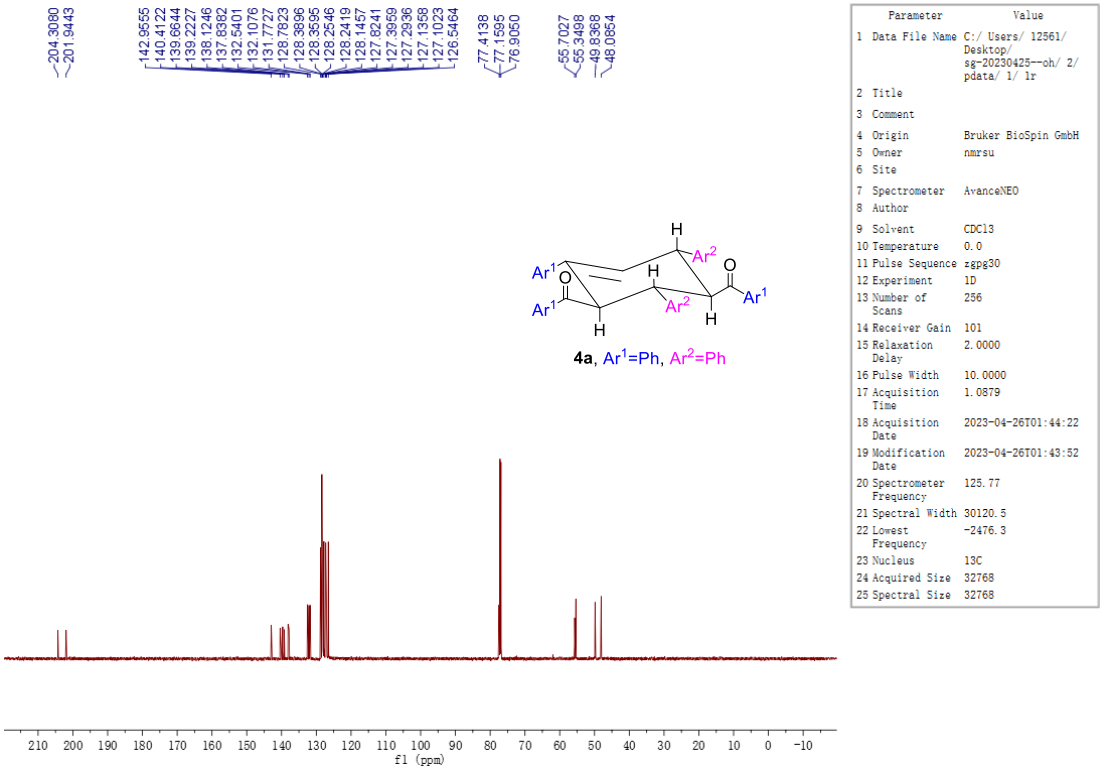
19	Modification Date	2023-04-26T01:29:26
20	Spectrometer Frequency	500.16
21	Spectral Width	10000.0
22	Lowest Frequency	-1932.1
23	Nucleus	1H
24	Acquired Size	32768
25	Spectral Size	65536

Figure S34 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **4a**



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6	Site	
7	Spectrometer	AvanceNEO
8	Author	
9	Solvent	CDCl3
10	Temperature	0.0
11	Pulse Sequence	zgpg30
12	Experiment	1D
13	Number of Scans	256
14	Receiver Gain	101
15	Relaxation Delay	2.0000
16	Pulse Width	10.0000
17	Acquisition Time	1.0879
18	Acquisition Date	2023-04-26T01:44:22
19	Modification Date	2023-04-26T01:43:52
20	Spectrometer Frequency	125.77
21	Spectral Width	30120.5
22	Lowest Frequency	-2476.3
23	Nucleus	13C
24	Acquired Size	32768
25	Spectral Size	32768

Figure S35 ^{13}C NMR (126 MHz, $CDCl_3$) of compound **4a**

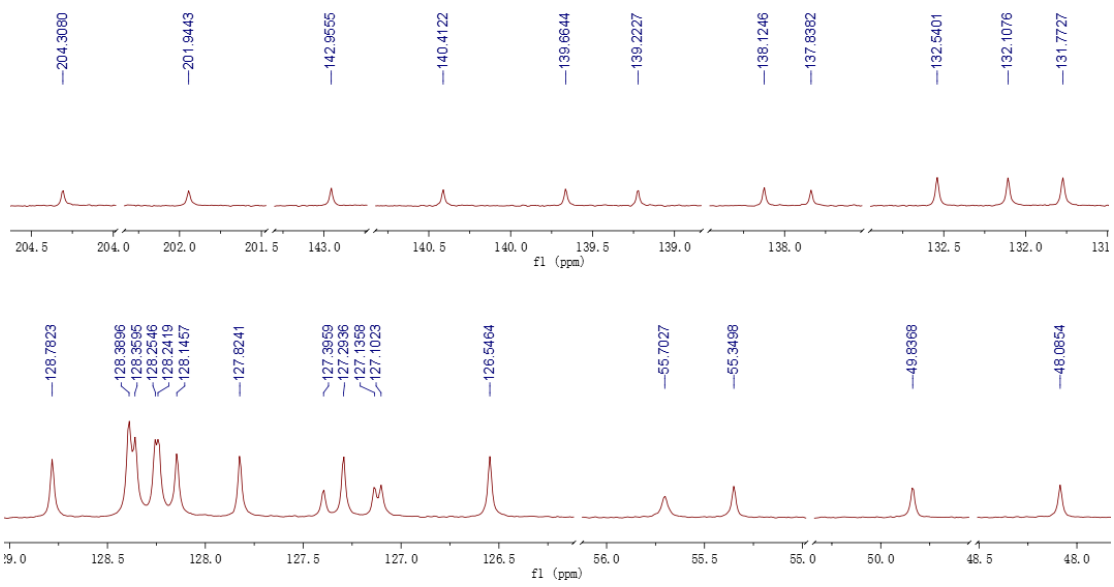
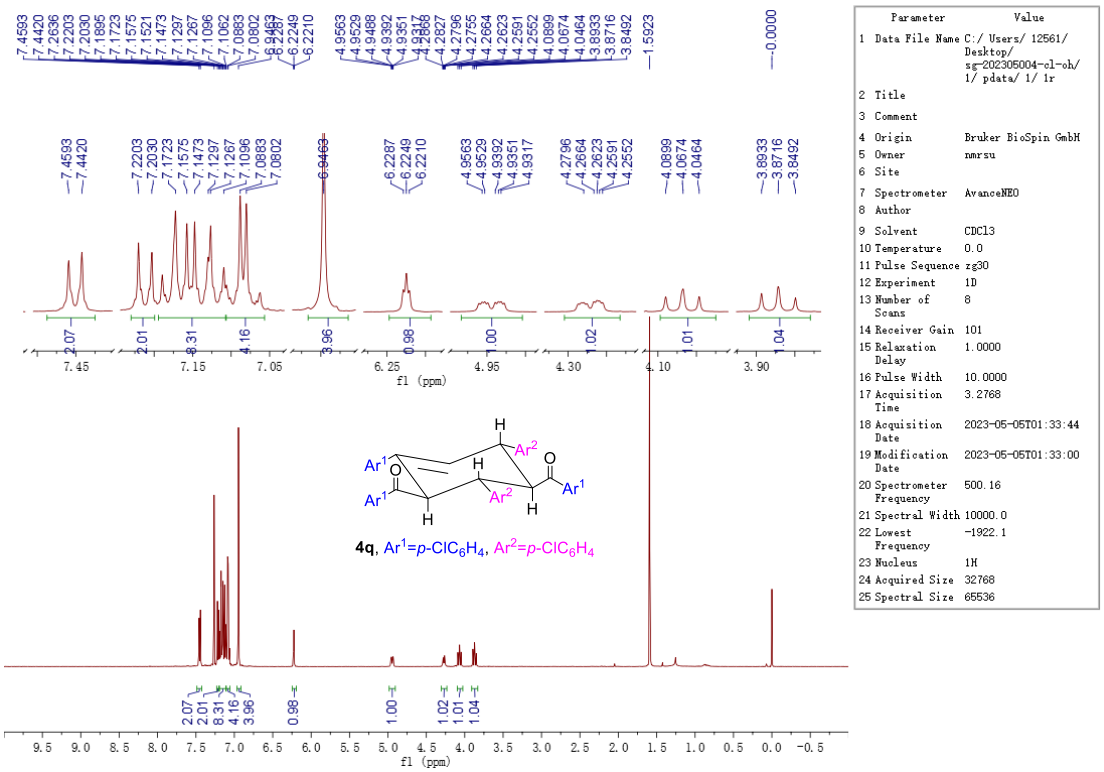


Figure S36 Expanded ^{13}C NMR (126 MHz, $CDCl_3$) of compound **4a**



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4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	AvanceNEO
8	Author	
9	Solvent	CDCl3
10	Temperature	0.0
11	Pulse Sequence	zg30
12	Experiment	1D
13	Number of Scans	8
14	Receiver Gain	101
15	Relaxation Delay	1.0000
16	Pulse Width	10.0000
17	Acquisition Time	3.2768
18	Acquisition Date	2023-05-05T01:33:44
19	Modification Date	2023-05-05T01:33:00
20	Spectrometer Frequency	500.16
21	Spectral Width	10000.0
22	Lowest Frequency	-1922.1
23	Nucleus	1H
24	Acquired Size	32768
25	Spectral Size	65536

Figure S37 ^{1}H NMR (500 MHz, $CDCl_3$) of compound **4q**