Supporting information

Visible light mediated, metal-free carbene transfer reactions of diazoalkanes with propargylic alcohols

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General Information

Unless otherwise noted, all commercially available compounds were used as provided without further purification. Chemicals used in this manuscript were purchased from Sigma Aldrich, Alfa Aesar, Fluorochem and Carl Roth.

Solvents used in reactions were p.A. grade. All reactions were performed under argon using degassed solvents. Solvents for chromatography were technical grade and distilled prior to use. Analytical thin-layer chromatography (TLC) was performed on Macherey-Nagel silica gel aluminium plates with F-254 indicator, visualised by irradiation with UV light. Column chromatography was performed using silica gel Merck 60 (particle size 0.063 - 0.2 mm). Solvent mixtures are understood as volume/volume.

¹H-NMR, ¹⁹F-NMR and ¹³C-NMR were recorded on a Varian AV600/AV400 or an Agilent DD2 400 NMR spectrometer in CDCl₃. Data are reported in the following order: chemical shift (δ) in ppm; multiplicities are indicated br (broadened singlet), s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet); coupling constants (*J*) are in Hertz (Hz).

HRMS data were recorded on a ThermoFisher Scientific LTQ Orbitrap XL using ESI ionization or on a Finnigan MAT 95 using EI ionization at 70 eV.

IR spectra were recorded on a Perkin Elmer-100 spectrometer and are reported in terms of frequency of absorption (cm⁻¹).

The following equipment was utilized for the addition of sodium nitrite: Syringe pump: Chemyx Inc. Model Fusion 710.

LEDs used in this manuscript were purchased from Conrad Electronics:

High Power LED-Module, 3 W, 30 lm, 30 °, 470 nm, art.nr. 180745 – 62.

Important safety note

NI

Safety hazards of diazo acetonitrile, described within this manuscript, have not been investigated. Handling of diazo compounds should only be done in a well-ventilated fume cupboard using an additional blast shield. No incidents occurred handling of diazoalkanes during the preparation of this manuscript, yet the reader should be aware of carcinogenicity and explosiveness of the herein described diazo compounds. General safety precautions when working with diazomethane and its derivatives should be followed. Any reactions described in this manuscript should not be performed without strict risk assessment and proper safety precautions.

Experimental Procedures

General procedure for the intercepted rearrangement reaction (GP)

In a test tube and under air, the substrate (1.0 eq.) and the diazoalkane (0.3 mmol, 4.0 eq.) were dissolved in 3.0 mL CHCl₃ and irradiated with one 3 W LED (distance 1.5 cm, cooling of the setup from the outside with a fan). and then stirred for another 11 h. The crude reaction mixture was purified by column chromatography using hexanes : EtOAc as eluent to afford the final product.

Overview on different alcohols tested

	$CO_2Me + R^OH - DCM$	→ O-H	insertion
entry	alcohol	solvent	yield
1	phenol	CHCl ₃	no reaction
2	cyclohexanol	CHCl ₃	no reaction
3	hexanol	CHCl ₃	no reaction
4	propargyl alcohol	CHCl ₃	no reaction
5	cinnamyl alcohol	CHCl ₃	no reaction
6	(E)-4-phenylbut-3-en-2-ol	CHCl ₃	no reaction
7	(E)-2-methyl-4-phenylbut-3-en-2-ol	CHCl ₃	sluggish reaction
Reaction were carried out according to GP.			

Physical data

methyl 2-(2-hydroxypropan-2-yl)-1-phenyl-3-(p-tolyl)cycloprop-2-ene-1-carboxylate (3a)



The title compound was synthesized according to the general procedure GP and was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as yellow oil (91%, 88 mg).

¹**H** NMR (600 MHz, Chloroform-*d*): $\delta = 7.54$ (s, 2H), 7.40 – 7.34 (m, 2H), 7.26 (ddd, J = 7.9, 6.4, 3.1 Hz, 4H), 7.22 – 7.16 (m, 1H), 3.74 (s, 3H), 3.05 (s, 1H), 2.40 (s, 3H), 1.63 (s, 3H), 1.47 (s, 3H) ppm.

¹³**C NMR** (151 MHz, Chloroform-*d*): $\delta = 175.5$, 140.5, 139.8, 129.9, 129.7 128.0, 127.9, 126.3, 122.8, 118.7, 107.1, 69.2, 52.2, 36.9, 29.7, 28.7, 21.5 ppm.

IR (**KBr**): 3430, 3027, 2978, 2871, 2586, 2296, 1867, 1714, 1604, 1501, 1441, 1369, 1281, 1214, 1018, 985, 948, 855, 821, 769, 704, 648, 556, 525, 498 cm⁻¹.

HRMS (ESI): mass found: 345.14578, mass calculated for $C_{21}H_{22}NaO_3^+$: 345.14612.

methyl 1-(4-ethoxyphenyl)-2-(2-hydroxypropan-2-yl)-3-(p-tolyl)cycloprop-2-ene-1carboxylate (3b)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as yellow oil (86%, 95 mg).

¹**H** NMR (400 MHz, Chloroform-*d*): δ = 7.56 – 7.45 (m, 2H), 7.30 – 7.13 (m, 4H), 6.83 – 6.64 (m, 2H), 3.97 (q, *J* = 6.9 Hz, 2H), 3.71 (s, 3H), 2.80 (s, 1H), 2.38 (s, 3H), 1.62 (s, 3H), 1.44 (s, 3H), 1.36 (t, *J* = 6.9 Hz, 3H) ppm. ¹³**C** NMR (101 MHz, Chloroform-*d*): δ = 175.7, 157.4, 139.7, 132.4, 129.8, 129.6, 128.9, 122.9, 119.0, 114.6, 114.0, 107.3, 69.1, 63.3, 52.2, 36.2, 29.7, 28.7, 21.5, 14.8 ppm.

IR (**KBr**): 3449, 2976, 2931, 2558, 2162, 2041, 1869, 1723, 1602, 1510, 1438, 1388, 1341, 1234, 1172, 1115, 1035, 920, 823, 738, 681 cm⁻¹.

HRMS (ESI): mass found: 389.17184, mass calculated for $C_{23}H_{26}NaO_4^+$: 389.17233.

methyl 1-(4-fluorophenyl)-2-(2-hydroxypropan-2-yl)-3-(p-tolyl)cycloprop-2-ene-1carboxylate (3c)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as yellow oil (82%, 84 mg).

¹**H NMR** (400 MHz, Chloroform-*d*): $\delta = 7.53 - 7.44$ (m, 2H), 7.34 - 7.28 (m, 2H), 7.25 - 7.20 (m, 2H), 6.97 - 6.84 (m, 2H), 3.71 (s, 3H), 2.78 (s, 1H), 2.38 (s, 3H), 1.61 (s, 3H), 1.45 (s, 3H) ppm.

¹³**C NMR** (101 MHz, Chloroform-*d*): $\delta = 175.2$, 162.6, 160.2, 139.9, 136.3 (d, J = 3.2 Hz), 129.8, 129.7, 129.5 (d, J = 7.5 Hz), 122.6, 118.5, 114.7 (d, J = 20.9 Hz), 107.1, 69.2, 52.2, 36.1, 29.6, 28.7, 21.5 ppm.

¹⁹**F** NMR (376 MHz, Chloroform-*d*): $\delta = -116.5$ ppm.

IR (**KBr**): 3469, 3031, 2924, 2856, 2321, 2190, 2053, 2003, 1881, 1708, 1603, 1506, 1430, 1365, 1267, 1219, 1133, 990, 951, 911, 817, 731, 674, 658 cm⁻¹.

HRMS (ESI): mass found: 363.13669, mass calculated for $C_{21}H_{21}FNaO_3^+$: 363.13669.

methyl 1-(4-chlorophenyl)-2-(2-hydroxypropan-2-yl)-3-(p-tolyl)cycloprop-2-ene-1carboxylate (3d)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as yellow oil (69%, 74 mg).

¹**H NMR** (400 MHz, Chloroform-*d*): $\delta = 7.53 - 7.42$ (m, 2H), 7.31 - 7.26 (m, 2H), 7.25 - 7.17 (m, 4H), 3.71 (s, 3H), 2.76 (s, 1H), 2.38 (s, 3H), 1.60 (s, 3H), 1.44 (s, 3H) ppm.

¹³**C** NMR (101 MHz, Chloroform-*d*): δ = 174.9, 140.0, 139.1, 132.0, 129.8, 129.7, 129.3, 128.1, 122.4, 106.8, 69.1, 52.3, 36.2, 29.6, 28.7, 21.5 ppm.

IR (**KBr**): 3886, 3442, 3028, 2924, 2856, 2732, 2582, 2491, 2400, 2293, 2249, 2173, 2100, 1868, 1716, 1607, 1570, 1492, 1454, 1375, 1283, 1218, 1091, 1013, 949, 900, 856, 823, 730, 677, 643, 563, 504, 479 cm⁻¹. **HRMS** (**FSD:** mass found: 370, 10600, mass calculated for *C*. **H**. (**IN**₂O, ⁺, 270, 10714)

HRMS (ESI): mass found: 379.10690, mass calculated for $C_{21}H_{21}CINaO_3^+$: 379.10714.

methyl 1-(4-bromophenyl)-2-(2-hydroxypropan-2-yl)-3-(p-tolyl)cycloprop-2-ene-1carboxylate (3e)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as yellow oil (76%, 92 mg).

¹**H NMR** (600 MHz, Chloroform-*d*): δ = 7.55 – 7.42 (m, 2H), 7.36 (d, *J* = 8.5 Hz, 2H), 7.24 (m, *J* = 7.6 Hz, 4H), 3.72 (s, 3H), 2.90 (s, 1H), 2.40 (s, 3H), 1.61 (s, 3H), 1.46 (s, 3H) ppm.

¹³C NMR (151 MHz, Chloroform-*d*): δ = 174.9, 140.1, 139.6, 131.1, 129.8, 129.7, 122.4, 120.1, 118.1, 106.7, 69.2, 52.3, 36.3, 29.6, 28.7, 21.5 ppm.

IR (**KBr**): 3884, 3779, 3442, 3028, 2978, 2732, 2584, 2491, 2403, 2293, 2249, 2175, 2097, 1903, 1868, 1715, 1609, 1487, 1440, 1383, 1210, 1074, 1009, 948, 902, 856, 823, 792, 729, 644, 593, 559, 501, 466 cm⁻¹. **HRMS (ESI):** mass found: 423.05673, mass calculated for $C_{21}H_{21}BrNaO_3^+$: 423.05663

TRMS (ESI): mass round. 425.03075, mass calculated for $C_{21}\pi_{21}BINaO_3$. 425.03005

methyl 1-(3-chlorophenyl)-2-(2-hydroxypropan-2-yl)-3-(p-tolyl)cycloprop-2-ene-1carboxylate (3f)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as yellow oil (69%, 78 mg).

¹**H NMR** (400 MHz, Chloroform-*d*): $\delta = 7.52 - 7.41$ (m, 2H), 7.35 (dt, J = 2.5, 1.1 Hz, 1H), 7.27 - 7.21 (m, 3H), 7.20 - 7.11 (m, 2H), 3.71 (s, 3H), 2.79 (s, 1H), 2.38 (s, 3H), 1.61 (s, 3H), 1.45 (s, 3H) ppm.

¹³**C NMR** (101 MHz, Chloroform-*d*): δ = 174.7, 142.7, 140.1, 133.8, 129.8, 129.2, 128.0, 126.5, 126.1, 122.3, 117.9, 106.8, 69.1, 52.3, 36.5, 29.6, 28.8, 21.5 ppm.

IR (**KBr**): 3980, 3877, 3735, 3471, 2921, 2853, 2733, 2635, 2586, 2517, 2354, 2296, 2249, 2177, 2109, 1909, 1868, 1719, 1595, 1569, 1506, 1469, 1383, 1205, 1030, 979, 949, 915, 883, 854, 819, 716, 649, 600, 565, 515 cm⁻¹.

HRMS (ESI): mass found: 379.10666, mass calculated for $C_{21}H_{21}CINaO_3^+$: 379.10714.

methyl 2-(2-hydroxypropan-2-yl)-1-(3-methoxyphenyl)-3-(p-tolyl)cycloprop-2-ene-1carboxylate (3g)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as yellow oil (74%, 78 mg).

¹**H NMR** (600 MHz, Chloroform-*d*): 7.51 (dd, J = 8.2, 1.9 Hz, 2H), 7.23 (d, J = 7.9 Hz, 2H), 7.17 (t, J = 7.9 Hz, 1H), 6.95 - 6.92 (m, 2H), 6.74 (ddd, J = 8.3, 2.5, 1.0 Hz, 1H), 3.75 (s, 3H), 3.73 (s, 3H), 2.90 (s, 1H), 2.39 (s, 3H), 1.65 (s. 3H), 1.47 (s. 3H) ppm.

¹³C NMR (151 MHz, Chloroform-*d*): $\delta = 175.3$, 159.3, 142.1, 139.8, 129.9, 129.7, 129.0, 122.7, 120.4, 118.3, 114.0, 111.4, 107.1, 69.1, 55.1, 52.2, 36.9, 29.7, 28.7, 21.5 ppm.

IR (KBr): 3432, 2975, 2161, 2031, 1864, 1713, 1606, 1491, 1441, 1350, 1229, 1113, 1032, 934, 863, 816, 711 cm⁻¹.

HRMS (ESI): mass found: 375.15613, mass calculated for $C_{22}H_{24}NaO_4^+$: 375.15668.

1-(2-fluorophenyl)-2-(2-hydroxypropan-2-yl)-3-(p-tolyl)cycloprop-2-ene-1methyl carboxylate (3h)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using n-hexane/EtOAc 20:1 as yellow oil (42%, 43 mg).

¹**H NMR** (400 MHz, Chloroform-*d*): $\delta = 7.71 - 7.43$ (m, 2H), 7.33 - 7.16 (m, 4H), 7.08 - 6.90 (m, 2H), 3.70 (s, 3H), 2.91 (s, 1H), 2.39 (s, 3H), 1.60 (s, 3H), 1.54 (s, 3H) ppm.

¹³C NMR (101 MHz, Chloroform-*d*): δ = 174.9, 162.9, 160.5, 139.9, 129.7 (d, *J* = 13.5 Hz), 129.5 (d, *J* = 4.3 Hz), 128.6 (d, J = 8.3 Hz), 124.0 (d, J = 3.3 Hz), 122.9, 119.0, 115.2, 115.0, 107.8, 69.1, 52.4, 29.2, 28.6, 21.5 ppm. ¹⁹**F NMR** (376 MHz, Chloroform-*d*): δ = -114.59 ppm.

IR (KBr): 3896, 3745, 3454, 2926, 2733, 2588, 2516, 2422, 2251, 2171, 2125, 2041, 1913, 1867, 1715, 1612, 1582, 1488, 1451, 1379, 1293, 1224, 996, 949, 910, 858, 823, 755, 678, 647, 600, 567, 538, 487, 470 cm⁻¹. **HRMS (ESI):** mass found: 363.13651, mass calculated for $C_{21}H_{21}FNaO_3^+$: 363.13669.

2-(2-hydroxypropan-2-yl)-1-(naphthalen-2-yl)-3-(p-tolyl)cycloprop-2-ene-1methyl carboxylate (3i)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as green oil (82%, 92 mg).

¹**H NMR** (400 MHz, Chloroform-*d*): $\delta = 7.86 - 7.65$ (m, 4H), 7.61 - 7.52 (m, 2H), 7.47 (dd, J = 8.5, 1.8 Hz, 1H), 7.43 – 7.34 (m, 2H), 7.28 – 7.22 (m, 2H), 3.75 (s, 3H), 2.90 (s, 1H), 2.40 (s, 3H), 1.65 (s, 3H), 1.45 (s, 3H)

ppm. ¹³**C NMR** (101 MHz, Chloroform-*d*): δ = 175.4, 139.9, 138.1, 133.3, 132.1, 129.9, 129.7, 127.7, 127.56, 127.52, 126.3, 126.2, 125.8, 125.4, 122.8, 118.8, 107.1, 69.2, 52.3, 37.1, 29.7, 29.6, 28.7, 21.5 ppm.

IR (KBr): 3430, 2931, 2728, 2489, 2293, 2162, 2031, 1866, 1711, 1602, 1508, 1445, 1366, 1238, 1172, 1027, 951, 901, 819, 752, 703 cm⁻¹

HRMS (ESI): mass found: 395.16133, mass calculated for $C_{25}H_{24}NaO_3^+$: 395.16177.

methyl 2-(2-hydroxypropan-2-yl)-1-(naphthalen-1-yl)-3-(p-tolyl)cycloprop-2-ene-1carboxylate (3j)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as yellow solid (71%, 80 mg).

¹**H** NMR (600 MHz, Chloroform-*d*): $\delta = 8.24$ (d, J = 8.5 Hz, 1H), 7.86 (dd, J = 8.2, 1.3 Hz, 1H), 7.75 (d, J = 8.1 Hz, 1H), 7.67 – 7.61 (m, 2H), 7.56 (ddd, J = 8.3, 6.7, 1.4 Hz, 1H), 7.49 (ddd, J = 8.1, 6.7, 1.2 Hz, 1H), 7.45 (dd, J = 7.1, 1.2 Hz, 1H), 7.35 – 7.24 (m, 3H), 3.64 (s, 3H), 3.18 (s, 1H), 2.44 (s, 3H), 1.72 (s, 3H), 1.36 (s, 3H) ppm. ¹³C NMR (151 MHz, Chloroform-*d*): $\delta = 177.0$, 139.9, 138.3, 133.6, 132.8, 129.8, 129.7, 128.6, 127.8, 126.1, 125.6, 125.4, 125.3, 124.8, 123.6, 121.3, 108.8, 69.4, 52.6, 36.1, 30.5, 28.1, 21.5 ppm.

IR (**KBr**): 3843, 3502, 3044, 2925, 2858, 2660, 2321, 2160, 2106, 2001, 1918, 1868, 1706, 1587, 1508, 1434, 1361, 1230, 1174, 1108, 1047, 971, 856, 775 cm⁻¹.

HRMS (ESI): mass found: 395.16132, mass calculated for $C_{25}H_{24}NaO_3^+$: 395.16177.

ethyl 2-(2-hydroxypropan-2-yl)-1-phenyl-3-(p-tolyl)cycloprop-2-ene-1-carboxylate (3k)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as yellow oil (78%, 79 mg).

¹**H** NMR (600 MHz, Chloroform-*d*): $\delta = 7.57 - 7.47$ (m, 2H), 7.42 - 7.32 (m, 2H), 7.25 (td, J = 7.6, 3.8 Hz, 4H), 7.22 - 7.16 (m, 1H), 4.22 (qq, J = 10.9, 7.1 Hz, 2H), 3.10 (s, 1H), 2.40 (s, 3H), 1.64 (s, 3H), 1.46 (s, 3H), 1.25 (t, J = 7.1 Hz, 3H) ppm.

¹³C NMR (151 MHz, Chloroform-*d*): $\delta = 174.9$, 140.6, 139.7, 129.9, 129.7, 128.0, 127.8, 126.2, 122.9, 118.7, 106.9, 69.1, 61.0, 37.1, 29.7, 28.8, 21.5, 14.3 ppm.

IR (**KBr**): 3778, 3439, 3055, 3026, 2979, 2928, 2734, 2490, 2296, 2065, 1955, 1837, 1805, 1712, 1604, 1501, 1449, 1379, 1210, 1028, 977, 951, 856, 821, 769, 703, 647, 595, 557, 524, 496 cm⁻¹.

HRMS (ESI): mass found: 359.16122, mass calculated for $C_{22}H_{24}NaO_3^+$: 359.16177.

methyl 2-(2-hydroxypropan-2-yl)-1,3-diphenylcycloprop-2-ene-1-carboxylate (31)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as colorless solid (81%, 75 mg).

¹**H NMR** (600 MHz, Chloroform-*d*): $\delta = 7.66 - 7.61$ (m, 2H), 7.52 - 7.33 (m, 5H), 7.30 - 7.23 (m, 2H), 7.24 - 7.17 (m, 1H), 3.74 (s, 3H), 3.04 (s, 1H), 1.64 (s, 3H), 1.48 (s, 3H) ppm.

¹³**C** NMR (151 MHz, Chloroform-*d*): $\delta = 175.4$, 140.3, 129.9, 129.5, 128.9, 128.1, 127.9, 126.4, 125.7, 119.9, 107.3, 69.2, 52.33, 52.31, 37.1, 29.6, 28.7 ppm.

IR (**KBr**): 3508, 3075, 3021, 2981, 2467, 2322, 2219, 2183, 2118, 2034, 1997, 1858, 1697, 1601, 1573, 1489, 1435, 1366, 1231, 1156, 1071, 983, 945, 855, 818, 765, 733, 696 cm⁻¹.

HRMS (ESI) : mass found: 331.13007, mass calculated for $C_{20}H_{20}NaO_3^+$: 331.13047

methyl 2-(4-ethylphenyl)-3-(2-hydroxypropan-2-yl)-1-phenylcycloprop-2-ene-1carboxylate (3m)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as yellow oil (76%, 76 mg).

¹**H NMR** (400 MHz, Chloroform-*d*): $\delta = 7.57 - 7.49$ (m, 2H), 7.39 - 7.31 (m, 2H), 7.28 - 7.20 (m, 4H), 7.22 - 7.13 (m, 1H), 3.72 (s, 3H), 2.90 (s, 1H), 2.68 (q, J = 7.6 Hz, 2H), 1.63 (s, 3H), 1.45 (s, 3H), 1.24 (t, J = 7.6 Hz, 3H) ppm.

¹³C NMR (101 MHz, Chloroform-*d*): $\delta = 175.4$, 146.1, 140.5, 130.0, 128.5, 128.0, 127.9, 126.3, 123.0, 118.7, 107.1, 69.1, 52.2, 36.9, 29.7, 28.8, 28.7, 15.4 ppm.

IR (**KBr**): 3432, 3055, 3026, 2972, 2873, 2503, 2314, 1867, 1716, 1603, 1500, 1445, 1370, 1278, 1214, 1018, 985, 949, 892, 838, 769, 703, 648, 541, 504 cm⁻¹.

HRMS (ESI): mass found: 375.13528, mass calculated for $C_{21}H_{22}KO_3^+$: 375.13570.

methyl 2-(4-(tert-butyl)phenyl)-3-(2-hydroxypropan-2-yl)-1-phenylcycloprop-2-ene-1carboxylate (3n)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as yellow oil (69%, 75 mg).

¹**H NMR** (400 MHz, Chloroform-*d*): $\delta = 7.59 - 7.53$ (m, 2H), 7.48 - 7.43 (m, 2H), 7.40 - 7.34 (m, 2H), 7.29 - 7.22 (m, 2H), 7.21 - 7.14 (m, 1H), 3.72 (s, 3H), 3.01 (s, 1H), 1.63 (s, 3H), 1.46 (s, 3H), 1.34 (s, 3H) ppm.

¹³C NMR (101 MHz, Chloroform-*d*): δ = 175.4, 152.9, 140.5, 129.7, 128.0, 127.9, 126.3, 125.9, 122.8, 118.8, 107.1, 69.1, 52.2, 37.0, 34.9, 31.1, 29.7, 28.7 ppm.

IR (**KBr**): 3438, 3058, 3028, 2964, 2870, 2493, 2248, 1867, 1717, 1603, 1498, 1457, 1366, 1269, 1213, 1110, 1019, 985, 948, 912, 839, 733, 702, 670, 644, 556, 508 cm⁻¹.

HRMS (ESI): mass found: 387.19339, mass calculated for $C_{24}H_{28}NaO_3^+$: 387.19307.

methyl 2-(4-fluorophenyl)-3-(2-hydroxypropan-2-yl)-1-phenylcycloprop-2-ene-1carboxylate (30)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as yellow oil (74%, 72 mg).

¹**H NMR** (400 MHz, Chloroform-*d*): δ = 7.64 – 7.50 (m, 2H), 7.37 – 7.30 (m, 2H), 7.29 – 7.16 (m, 3H), 7.11 (t, *J* = 8.7 Hz, 2H), 3.73 (s, 3H), 2.92 (s, 1H), 1.59 (s, 3H), 1.46 (s, 3H) ppm.

¹³**C NMR** (101 MHz, Chloroform-*d*): δ = 175.1, 164.5, 162.0, 140.1, 131.8 (d, *J* = 8.9 Hz), 128.1, 127.7, 126.4, 122.0 (d, *J* = 3.3 Hz), 119.4 (d, *J* = 2.8 Hz), 116.3, 116.1, 106.4, 69.2, 52.3, 37.1, 29.6, 28.7 ppm.

¹⁹**F NMR** (376 MHz, Chloroform-*d*): δ = -109.9 ppm.

IR (**KBr**): 3470, 3058, 3028, 2926, 2856, 2159, 2009, 1939, 1882, 1707, 1598, 1499, 1437, 1369, 1218, 1183, 1151, 1096, 992, 951, 859, 833, 807, 766, 699 cm⁻¹.

HRMS (ESI): mass found: 349.12015, mass calculated for $C_{20}H_{19}FNaO_3^+$: 349.12104.

methyl 2-(2-hydroxypropan-2-yl)-3-(4-iodophenyl)-1-phenylcycloprop-2-ene-1carboxylate (3p)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as yellow oil (66%, 86 mg).

¹**H NMR** (600 MHz, Chloroform-*d*): $\delta = 7.84 - 7.72$ (m, 2H), 7.39 - 7.29 (m, 4H), 7.29 - 7.24 (m, 2H), 7.22 - 7.17 (m, 1H), 3.73 (s, 3H), 2.99 (s, 1H), 1.58 (s, 3H), 1.47 (s, 3H) ppm.

¹³C NMR (151 MHz, Chloroform-*d*): δ = 175.0, 139.9, 138.2, 131.3, 128.1, 127.8, 126.5, 125.2, 121.2, 106.6, 95.7, 69.4, 52.3, 37.1, 29.5, 28.7 ppm.

IR (**KBr**): 3559, 3462, 3055, 2975, 2931, 2491, 2322, 2191, 2165, 2092, 2011, 1969, 1874, 1706, 1581, 1550, 1478, 1428, 1390, 1363, 1277, 1226, 1181, 1134, 1054, 985, 950, 888, 860, 823, 794, 771, 738, 699, 661 cm⁻¹. **HRMS (ESI):** mass found: 457.02689, mass calculated for $C_{20}H_{19}INaO_3^+$: 457.02711.

methyl 2-([1,1'-biphenyl]-4-yl)-3-(2-hydroxypropan-2-yl)-1-phenylcycloprop-2-ene-1carboxylate (3q)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as yellow oil (59%, 68 mg).

¹**H NMR** (600 MHz, Chloroform-*d*): $\delta = 7.76 - 7.71$ (m, 2H), 7.71 - 7.66 (m, 2H), 7.65 - 7.60 (m, 2H), 7.52 - 7.35 (m, 5H), 7.33 - 7.27 (m, 2H), 7.24 (qt, J = 6.5, 1.4 Hz, 1H), 3.78 (s, 3H), 1.67 (s, 3H), 1.53 (s, 3H) ppm.

¹³**C NMR** (151 MHz, Chloroform-*d*): δ = 175.4, 142.3, 140.3, 140.2, 133.4, 130.4, 130.1, 128.9, 128.4, 128.1, 127.9, 127.8, 127.7, 127.1, 126.4, 124.6, 120.1, 107.0, 69.3, 52.3, 37.1, 29.7, 28.8 ppm.

IR (**KBr**): 3451, 3029, 2927, 2855, 2663, 2328, 2162, 2091, 1993, 1951, 1917, 1853, 1719, 1687, 1601, 1520, 1485, 1440, 1385, 1366, 1237, 1156, 1072, 981, 937, 845, 767, 696, 665 cm⁻¹.

HRMS (ESI): mass found: 407.16177, mass calculated for $C_{26}H_{24}NaO_3^+$: 407.16177.

methyl 2-(4-cyanophenyl)-3-(2-hydroxypropan-2-yl)-1-phenylcycloprop-2-ene-1carboxylate (3r)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as yellow solid (68%, 68 mg).

¹**H NMR** (600 MHz, Chloroform-*d*): $\delta = 7.86 - 7.62$ (m, 4H), 7.31 (d, J = 6.9 Hz, 2H), 7.27 (t, J = 7.5 Hz, 2H), 7.24 - 7.19 (m, 1H), 3.74 (s, 3H), 2.96 (s, 1H), 1.56 (s, 3H), 1.51 (s, 3H) ppm.

¹³C NMR (151 MHz, Chloroform-*d*): δ = 174.5, 139.3, 132.7, 130.3, 130.2, 128.3, 127.7, 126.8, 118.4, 112.6, 106.3, 69.7, 52.5, 52.4, 37.5, 29.4, 28.7 ppm.

IR (**KBr**): 3470, 3056, 2979, 2934, 2567, 2481, 2287, 2224, 2076, 2038, 2006, 1971, 1937, 1869, 1708, 1602, 1548, 1495, 1429, 1407, 1368, 1283, 1238, 1184, 1131, 1070, 988, 953, 861, 839, 796, 765, 701, 672 cm⁻¹. **HRMS (ESI):** mass found: 356.12518, mass calculated for $C_{21}H_{19}NNaO_3^+$: 356.12571.

methyl 2-(4-acetylphenyl)-3-(2-hydroxypropan-2-yl)-1-phenylcycloprop-2-ene-1carboxylate (3s)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as yellow solid (64%, 67 mg).

¹**H NMR** (400 MHz, Chloroform-*d*): $\delta = 8.04 - 7.95$ (m, 2H), 7.73 - 7.64 (m, 2H), 7.34 - 7.29 (m, 2H), 7.28 - 7.22 (m, 2H), 7.22 - 7.16 (m, 1H), 3.73 (s, 3H), 2.89 (s, 1H), 2.60 (s, 3H), 1.59 (s, 3H), 1.49 (s, 3H) ppm.

¹³C NMR (101 MHz, Chloroform-*d*): δ = 197.2, 174.8, 139.7, 137.2, 130.2, 129.9, 128.9, 128.2, 127.8, 126.6, 123.5, 106.7, 69.5, 52.3, 37.3, 29.5, 28.7, 26.6 ppm.

IR (**KBr**): 3779, 3454, 2976, 2648, 2504, 2323, 2196, 2162, 2121, 2055, 1982, 1951, 1869, 1801, 1703, 1677, 1600, 1559, 1494, 1432, 1405, 1359, 1233, 1182, 1133, 1072, 984, 953, 843, 794, 760, 731, 701, 670 cm⁻¹. **HRMS (ESI):** mass found: 373.14056, mass calculated for $C_{22}H_{22}NaO_4^+$: 373.14103.

methyl 2-(2-hydroxypropan-2-yl)-1-phenyl-3-(4-(trifluoromethoxy)phenyl)cycloprop-2-ene-1-carboxylate (3t)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as white solid (71%, 84 mg).

¹**H NMR** (600 MHz, Chloroform-*d*): $\delta = 7.69 - 7.60$ (m, 2H), 7.37 - 7.32 (m, 2H), 7.30 - 7.24 (m, 4H), 7.24 - 7.19 (m, 1H), 3.74 (s, 3H), 2.97 (s, 1H), 1.60 (s, 3H), 1.49 (s, 3H) ppm.

¹³C NMR (151 MHz, Chloroform-*d*): δ = 175.0, 149.7, 139.9, 131.4, 127.9 (d, *J* = 59.7 Hz), 126.5, 124.5, 121.4, 121.0, 106.2, 69.4, 52.3 (d, *J* = 4.2 Hz), 37.2, 29.5, 28.7 ppm.

¹⁹**F** NMR (376 MHz, Chloroform-*d*): $\delta = -57.7$ ppm.

IR (**KBr**): 3457, 3057, 2980, 2481, 2147, 2077, 1974, 1876, 1705, 1600, 1499, 1427, 1364, 1224, 1152, 984, 952, 921, 854, 807, 772, 741, 701, 659 cm⁻¹.

HRMS (ESI): mass found: 415.11209, mass calculated for $C_{21}H_{19}F_3NaO_4^+$: 415.11276.

methyl 2-(2-hydroxypropan-2-yl)-1-phenyl-3-(4-(trifluoromethyl)phenyl)cycloprop-2ene-1-carboxylate (3u)



F₃C

The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as white solid (83%, 93 mg).

¹**H NMR** (400 MHz, Chloroform-*d*): $\delta = 7.75 - 7.70$ (m, 2H), 7.69 - 7.63 (m, 2H), 7.36 - 7.31 (m, 2H), 7.29 - 7.23 (m, 2H), 7.23 - 7.17 (m, 1H), 3.73 (s, 3H), 3.17 (s, 1H), 1.58 (s, 3H), 1.50 (s, 3H) ppm.

¹³C NMR (101 MHz, Chloroform-*d*): δ = 174.8, 139.6, 131.2, 130.9, 130.0, 129.3, 128.2, 127.7, 126.6, 125.9 (q, *J* = 3.8 Hz), 123.2, 106.4, 69.5, 52.3, 37.3, 30.2, 29.4, 28.6 ppm.

¹⁹**F** NMR (282 MHz, Chloroform-*d*): $\delta = -62.8$ ppm.

IR (**KBr**): 3461, 3063, 2959, 2926, 2856, 2646, 2323, 2227, 2188, 2094, 1974, 1928, 1873, 1704, 1611, 1495, 1431, 1409, 1366, 1320, 1235, 1184, 1153, 1113, 1062, 986, 953, 842, 796, 767, 699, 662 cm⁻¹.

HRMS (ESI): mass found: 376.12815, mass calculated for $C_{21}H_{19}F_3O_3^+$: 376.12808.

methyl 2-(2-hydroxypropan-2-yl)-3-(3-methoxyphenyl)-1-phenylcycloprop-2-ene-1carboxylate (3v)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as yellow oil (60%, 61 mg).

¹**H NMR** (600 MHz, Chloroform-*d*): $\delta = 7.40 - 7.32$ (m, 3H), 7.29 - 7.17 (m, 4H), 7.15 (dd, J = 2.7, 1.5 Hz, 1H), 6.95 (ddd, J = 8.4, 2.7, 1.1 Hz, 1H), 3.82 (s, 3H), 3.74 (s, 3H), 2.95 (s, 1H), 1.63 (s, 3H), 1.47 (s, 3H) ppm. ¹³C NMR (151 MHz, Chloroform-*d*): δ = 175.3, 159.8, 140.3, 130.0, 128.1, 127.9, 126.9, 126.4, 122.4, 120.3,

115.3, 115.0, 107.2, 69.2, 55.3, 52.3, 37.2, 29.6, 28.7 ppm.

IR (KBr): 3442, 3058, 2977, 2839, 2597, 2503, 2091, 1947, 1867, 1716, 1596, 1487, 1433, 1368, 1321, 1286, 1223, 1076, 1042, 996, 954, 849, 787, 699, 659, 586, 509 cm⁻¹.

HRMS (ESI): mass found: 361.14099, mass calculated for $C_{21}H_{22}NaO_4^+$: 361.14103.

methyl 2-(2-hydroxypropan-2-yl)-1-phenyl-3-(m-tolyl)cycloprop-2-ene-1-carboxylate (3w)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using n-hexane/EtOAc 20:1 as yellow gel (84%, 82 mg).

¹**H NMR** (400 MHz, Chloroform-*d*): $\delta = 7.42$ (d, J = 7.4 Hz, 2H), 7.37 - 7.28 (m, 3H), 7.28 - 7.15 (m, 4H), 3.73(s, 3H), 2.89 (s, 1H), 2.37 (s, 3H), 1.62 (s, 3H), 1.45 (s, 3H) ppm. ¹³C NMR (101 MHz, Chloroform-*d*): $\delta = 175.4$, 140.4, 138.7, 130.3, 128.8, 128.0, 127.9, 127.1, 126.3, 125.6,

119.6, 107.2, 69.2, 52.2, 37.0, 29.7, 28.7, 21.3 ppm.

IR (KBr): 3432, 3055, 3026, 2978, 2341, 1950, 1869, 1716, 1601, 1489, 1442, 1369, 1281, 1214, 1018, 954, 913, 847, 789, 700, 657, 609, 552, 508 cm⁻¹.

HRMS (ESI): mass found: 345.14609, mass calculated for $C_{21}H_{22}NaO_3^+$: 345.14612.

2-(2-fluorophenyl)-3-(2-hydroxypropan-2-yl)-1-phenylcycloprop-2-ene-1methvl carboxylate (3x)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using n-hexane/EtOAc 20:1 as yellow gel (77%, 75 mg).

¹**H NMR** (600 MHz, Chloroform-*d*): $\delta = 7.52$ (td, J = 7.3, 1.7 Hz, 1H), 7.42 – 7.33 (m, 3H), 7.27 (ddd, J = 7.8, 6.4, 1.2 Hz, 2H), 7.23 - 7.14 (m, 3H), 3.75 (s, 3H), 2.83 (s, 1H), 1.60 (s, 3H), 1.50 (s, 3H) ppm.

¹³C NMR (151 MHz, Chloroform-*d*): δ = 175.06, 161.71, 160.03, 140.08, 131.43 (d, *J* = 8.2 Hz), 131.23 (d, *J* = 2.4 Hz), 128.14, 127.94, 126.51, 124.63 (d, J = 3.6 Hz), 121.69 (d, J = 3.1 Hz), 115.96 (d, J = 20.6 Hz), 114.44 (d, J = 14.8 Hz), 101.42, 69.81, 52.33, 35.99, 29.43 (d, J = 2.9 Hz), 28.83 ppm.

¹⁹**F** NMR (564 MHz, Chloroform-*d*): $\delta = -111.2$ ppm.

IR (KBr): 3648, 3443, 3065, 2924, 2855, 2325, 2212, 2162, 2083, 2010, 1957, 1927, 1875, 1701, 1605, 1579, 1492, 1446, 1372, 1292, 1263, 1208, 1178, 1100, 1019, 978, 949, 913, 893, 858, 830, 796, 757, 700, 657 cm⁻¹. **HRMS (ESI):** mass found: 349.12042, mass calculated for $C_{20}H_{19}FNaO_3^+$: 349.12104.

methyl 2-(2-hydroxypropan-2-yl)-1-phenyl-3-(o-tolyl)cycloprop-2-ene-1-carboxylate (3y)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as yellow solid (67%, 65 mg).

¹**H NMR** (400 MHz, Chloroform-*d*): $\delta = 7.55$ (dd, J = 7.5, 1.5 Hz, 1H), 7.37 – 7.07 (m, 8H), 3.74 (s, 3H), 2.80 (s, 1H), 2.49 (s, 3H), 1.54 (s, 3H), 1.48 (s, 3H) ppm.

¹³C NMR (101 MHz, Chloroform-*d*): $\delta = 175.6$, 140.8, 138.1, 131.3, 130.7, 129.5, 128.0, 127.8, 126.2, 126.1, 125.3, 119.5, 105.3, 69.9, 52.2, 36.6, 29.9, 29.2, 20.5 ppm.

IR (**KBr**): 3445, 3059, 3028, 2973, 2926, 2855, 2494, 2333, 2159, 2088, 2010, 1930, 1859, 1695, 1600, 1492, 1440, 1373, 1293, 1204, 1172, 1089, 1041, 1022, 972, 913, 894, 855, 824, 793, 761, 699, 657 cm⁻¹. **HRMS (ESI):** mass found: 345.14584, mass calculated for $C_{21}H_{22}NaO_3^+$: 345.14612.

methyl 2-(2-hydroxypropan-2-yl)-3-(2-methoxyphenyl)-1-phenylcycloprop-2-ene-1carboxylate (3z)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as yellow oil (94%, 96 mg).

¹**H NMR** (400 MHz, Chloroform-*d*): $\delta = 7.46 - 7.30$ (m, 4H), 7.26 - 7.20 (m, 2H), 7.19 - 7.12 (m, 1H), 7.07 - 6.91 (m, 2H), 3.96 (s, 3H), 3.71 (s, 3H), 1.53 (s, 3H), 1.39 (s, 3H) ppm.

¹³**C NMR** (101 MHz, Chloroform-*d*): δ = 174.8, 157.4, 140.3, 131.1, 131.0, 127.9, 126.2, 121.3, 120.5, 114.8, 110.7, 102.4, 70.1, 55.5, 52.0, 36.0, 29.4, 29.0 ppm.

IR (**KBr**): 3450, 3056, 2993, 2935, 2496, 2329, 2162, 2082, 2037, 1953, 1913, 1872, 1695, 1596, 1490, 1439, 1372, 1266, 1180, 1094, 1019, 977, 891, 857, 819, 791, 753, 701, 656 cm⁻¹.

HRMS (ESI): mass found: 361.14056, mass calculated for $C_{21}H_{22}NaO_4^+$: 361.14103.

methyl 2-(2-hydroxypropan-2-yl)-1-phenyl-3-(thiophen-2-yl)cycloprop-2-ene-1carboxylate (3aa)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as orange solid (48%, 45 mg).

¹**H NMR** (400 MHz, Chloroform-*d*): δ = 7.49 (dd, *J* = 5.1, 1.2 Hz, 1H), 7.43 – 7.37 (m, 2H), 7.31 – 7.15 (m, 4H), 7.08 (dd, *J* = 5.1, 3.7 Hz, 1H), 3.72 (s, 3H), 2.91 (s, 1H), 1.63 (s, 3H), 1.47 (s, 3H).

ppm. ¹³C NMR (101 MHz, Chloroform-*d*): $\delta = 174.7$, 140.0, 129.9, 129.8, 128.1, 128.0, 127.9, 127.8, 126.6, 117.4, 102.1, 68.9, 52.3, 38.5, 29.4, 28.7 ppm.

IR (**KBr**): 3492, 3090, 2956, 2328, 2186, 2011, 1859, 1701, 1599, 1493, 1435, 1367, 1227, 1152, 1075, 995, 964, 941, 847, 792, 768, 728, 698, 666 cm⁻¹.

HRMS (ESI): mass found: 337.08731, mass calculated for $C_{18}H_{18}SNaO_3^+$: 337.08689.

methyl 2-(3,5-bis(trifluoromethyl)phenyl)-3-(2-hydroxypropan-2-yl)-1-phenylcycloprop-2-ene-1-carboxylate (3ab)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as yellow oil (61%, 81 mg).

¹**H NMR** (400 MHz, Chloroform-*d*): $\delta = 8.10 - 7.97$ (m, 2H), 7.89 - 7.81 (m, 1H), 7.35 - 7.14 (m, 5H), 3.75 (s, 3H), 2.93 (s, 1H), 1.53 (s, 3H), 1.52 (s, 3H) ppm.

¹³**C** NMR (101 MHz, Chloroform-*d*): $\delta = 174.2$, 139.0, 132.5 (q, J = 33.8 Hz), 129.3, 128.4, 128.3, 127.6, 126.9, 125.2, 124.2, 122.7 (dt, J = 7.5, 3.8 Hz), 121.5, 105.6, 69.8, 52.4, 37.8, 29.6, 29.2, 28.6 ppm.

¹⁹**FNMR** (282 MHz, Chloroform-*d*): $\delta = -63.0$ ppm.

IR (**KBr**): 3446, 3062, 3030, 2980, 2858, 2556, 2252, 2103, 1949, 1865, 1804, 1722, 1610, 1494, 1453, 1377, 1280, 1014, 956, 904, 840, 766, 731, 702, 610, 508 cm⁻¹.

HRMS (ESI): mass found: 444.11532, mass calculated for $C_{22}H_{18}F_6O_3^+$: 444.11547.

methyl 2-(1-hydroxycyclobutyl)-1-phenyl-3-(p-tolyl)cycloprop-2-ene-1-carboxylate (3ac)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as yellow gel (43%, 42 mg).

¹**H NMR** (600 MHz, Chloroform-*d*): $\delta = 7.71 - 7.61$ (m, 2H), 7.49 - 7.34 (m, 5H), 7.29 - 7.18 (m, 3H), 3.75 (s, 3H), 3.22 (s, 1H), 2.51 (ddt, J = 11.8, 8.0, 3.5 Hz, 1H), 2.45 - 2.28 (m, 3H), 1.86 - 1.70 (m, 2H) ppm.

¹³**C** NMR (151 MHz, Chloroform-*d*): δ = 175.4, 140.0, 129.9, 129.4, 129.0, 128.1, 127.8, 126.5, 125.8, 118.1, 107.4, 72.4, 52.3, 37.1, 36.4, 12.6 ppm.

IR (**KBr**): 3439, 3058, 3025, 2989, 2948, 2849, 2595, 2339, 2249, 1956, 1869, 1716, 1600, 1492, 1441, 1382, 1214, 1116, 1072, 1017, 916, 826, 763, 730, 697, 642, 588, 496 cm⁻¹.

HRMS (ESI): mass found: 343.13046, mass calculated for $C_{21}H_{20}NaO_3^+$: 343.13047.

methyl 2-(1-hydroxycyclopentyl)-1-phenyl-3-(p-tolyl)cycloprop-2-ene-1-carboxylate (3ad)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as yellow oil (62%, 62 mg).

¹**H NMR** (600 MHz, Chloroform-*d*): δ = 7.65 – 7.58 (m, 2H), 7.46 – 7.41 (m, 2H), 7.41 – 7.34 (m, 3H), 7.28 – 7.24 (m, 2H), 7.23 – 7.17 (m, 1H), 3.74 (s, 3H), 2.66 (s, 1H), 2.10 – 1.96 (m, 2H), 1.95 – 1.82 (m, 4H), 1.79 – 1.59 (m, 2H) ppm.

¹³C NMR (151 MHz, Chloroform-*d*): δ = 175.3, 140.3, 129.8, 129.3, 128.9, 128.1, 127.9, 126.4, 125.9, 119.1, 107.4, 79.3, 52.2, 40.4, 39.7, 37.1, 23.54, 23.52 ppm.

IR (**KBr**): 3439, 3058, 3026, 2955, 2873, 2596, 2338, 2248, 1956, 1870, 1715, 1600, 1491, 1441, 1379, 1216, 1071, 1013, 917, 818, 763, 731, 697, 661, 597, 532 cm⁻¹.

HRMS (ESI): mass found: 357.14606, mass calculated for $C_{22}H_{22}NaO_3^+$: 357.14612.

methyl 2-(1-hydroxycyclohexyl)-1-phenyl-3-(p-tolyl)cycloprop-2-ene-1-carboxylate (3ae)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as yellow gel (49%, 52 mg).

¹**H NMR** (600 MHz, Chloroform-*d*): δ = 7.66 – 7.62 (m, 2H), 7.47 – 7.38 (m, 3H), 7.38 – 7.33 (m, 2H), 7.28 – 7.23 (m, 2H), 7.22 – 7.16 (m, 1H), 3.75 (s, 3H), 3.04 (s, 1H), 2.12 – 2.06 (m, 1H), 1.94 – 1.85 (m, 1H), 1.82 – 1.75 (m, 2H), 1.74 – 1.64 (m, 1H), 1.58 – 1.54 (m, 2H), 1.45 – 1.21 (m, 2H), ppm.

¹³C NMR (151 MHz, Chloroform-*d*): δ = 175.7, 140.4, 130.0, 129.5, 129.0, 128.0, 127.9, 126.3, 125.8, 119.7, 107.6, 71.1, 52.3, 38.1, 37.3, 36.4, 25.1, 22.4, 22.1 ppm.

IR (**KBr**): 3444, 3058, 3027, 2936, 2856, 2664, 2597, 2509, 2337, 2248, 2094, 1954, 1863, 1714, 1600, 1491, 1443, 1387, 1342, 1219, 1157, 1065, 1022, 969, 911, 847, 823, 763, 731, 697, 654, 591, 530, 494 cm⁻¹. **HRMS (ESI):** mass found: 371.16156, mass calculated for $C_{23}H_{24}NaO_3^+$: 371.16177.

methyl 2-(1-hydroxy-1-phenylethyl)-1-phenyl-3-(p-tolyl)cycloprop-2-ene-1carboxylate (3af)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as yellow solid (99%, 121 mg).

¹**H** NMR (600 MHz, Chloroform-*d*): δ = 7.66 – 7.55 (m, 2H), 7.51 – 7.47 (m, 2H), 7.46 – 7.27 (m, 16H), 7.27 – 7.21 (m, 6H), 7.20 – 7.12 (m, 5H), 3.86 (s, 1H), 3.74 (s, 3H), 3.70 (s, 3H), 3.37 (s, 1H), 1.99 (s, 3H), 1.70 (s, 3H) ppm. ¹³**C** NMR (151 MHz, Chloroform-*d*): δ = 175.6, 175.4, 145.5, 144.4, 140.0, 139.7, 130.2, 130.1, 129.7, 129.6,

¹³C NMR (151 MHz, Chloroform-*d*): δ = 175.6, 175.4, 145.5, 144.4, 140.0, 139.7, 130.2, 130.1, 129.7, 129.6, 128.9, 128.8, 128.4, 128.2, 127.9, 127.7, 127.5, 127.3, 126.5, 126.4, 125.5, 125.1, 125.0, 119.7, 119.4, 109.6, 108.0, 72.7, 72.3, 52.5, 52.3, 37.6, 37.4, 30.6, 28.7 ppm.

IR (**KBr**): 3470, 3059, 3028, 2951, 2846, 2594, 2252, 1960, 1863, 1732, 1605, 1576, 1492, 1442, 1327, 1274 1218, 1102, 1059, 1016, 914, 826, 764, 731, 696, 626, 586, 543, 464 cm⁻¹.

HRMS (ESI): mass found: 393.14581, mass calculated for $C_{25}H_{22}NaO_3^+$: 393.14612.

methyl 2-(1-hydroxyethyl)-1,3-diphenylcycloprop-2-ene-1-carboxylate (3ag)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as yellow oil (53%, 47 mg).

¹**H** NMR (400 MHz, Chloroform-*d*): $\delta = 7.63 - 7.57$ (m, 3H), 7.45 - 7.32 (m, 10H), 7.29 - 7.15 (m, 6H), 5.14 (q, *J* = 6.6 Hz, 1H), 4.99 (q, *J* = 6.6 Hz, 1H), 3.72 (d, *J* = 3.2 Hz, 3H), 2.79 (s, 3H), 2.63 (s, 1H), 1.58 (d, *J* = 6.6 Hz, 3H), 1.48 (d, *J* = 6.6 Hz, 3H) ppm.

¹³**C NMR** (101 MHz, Chloroform-*d*): δ = 175.4, 175.1, 140.5, 140.3, 129.86, 129.83, 129.5, 129.4, 128.9, 128.1, 128.0, 126.5, 125.7, 117.0, 116.6, 109.4, 63.4, 63.1, 52.3, 52.2, 36.7, 22.6, 21.9 ppm.

IR (**KBr**): 3432, 3058, 3027, 2951, 2860, 2648, 2494, 2333, 2172, 2094, 1957, 1872, 1814, 1716, 1600, 1491, 1443, 1372, 1215, 1108, 1071, 1041, 917, 891, 820, 763, 698, 661, 488 cm⁻¹.

HRMS (ESI): mass found: 317.11475, mass calculated for $C_{19}H_{18}NaO_3^+$: 317.11482.

methyl 2-phenyl-2-((3-(p-tolyl)prop-2-yn-1-yl)oxy)acetate (4)



The title compound was synthesized according to the general procedure GP. And was obtained after silica gel column chromatography using *n*-hexane/EtOAc 20:1 as yellow oil (54%, 47 mg).

¹**H NMR** (600 MHz, Chloroform-*d*): $\delta = 7.52 - 7.44$ (m, 2H), 7.43 - 7.31 (m, 5H), 7.13 (d, *J* = 7.8 Hz, 2H), 5.29 (s, 1H), 4.53 (d, *J* = 16.0 Hz, 1H), 4.37 (d, *J* = 16.0 Hz, 1H), 3.73 (s, 3H), 2.35 (s, 3H) ppm. ¹³**C NMR** (151 MHz, Chloroform-*d*): $\delta = 170.9$, 138.8, 135.6, 131.7, 129.0, 128.9, 128.7, 127.6, 119.2, 87.5,

¹³C NMR (151 MHz, Chloroform-*d*): δ = 170.9, 138.8, 135.6, 131.7, 129.0, 128.9, 128.7, 127.6, 119.2, 87.5, 83.0, 78.7, 57.1, 52.4, 29.7, 21.5 ppm.

IR (**KBr**): 3857, 3498, 3030, 2922, 2856, 2332, 2233, 2164, 2089, 2011, 1906, 1743, 1605, 1509, 1447, 1353, 1259, 1211, 1174, 1097, 1023, 943, 817, 784, 733, 698 cm⁻¹.

HRMS (ESI): mass found: 317.11487, mass calculated for C₁₉H₁₈NaO₃⁺: 317.11482.

methyl 2-(2-methoxypropan-2-yl)-1-phenyl-3-(p-tolyl)cycloprop-2-ene-1-carboxylate (5)



To a solution of **3a** (64.5 mg, 0.2 mmol) in THF (2.0 mL) was added NaH (16.3 mg, 0.68 mmol) and MeI (91.8 mg, 0.64 mmol) at room temperature, and the mixture was stirred for 15 h at room temperature. H₂O (3 mL) was added to the reaction mixture followed by Extracted with EtOAc (3 mL \times 3), the organic layer was dried with MgSO₄. The compound was obtained after silica gel column chromatography using *n*-hexane/EtOAc 40:1–20:1 as yellow oil (53%, 36 mg).

¹**H NMR** (600 MHz, Chloroform-*d*): $\delta = 7.60 - 7.50$ (m, 2H), 7.45 - 7.37 (m, 2H), 7.26 (t, J = 8.0 Hz, 4H), 7.21 - 7.15 (m, 1H), 3.71 (s, 3H), 3.22 (s, 3H), 2.41 (s, 3H), 1.57 (s, 3H), 1.40 (s, 3H) ppm.

¹³**C NMR** (151 MHz, Chloroform-*d*): δ = 174.8, 140.7, 139.7, 129.8, 129.7, 128.2, 127.9, 126.2, 123.1, 117.2, 109.2, 74.6, 51.9, 51.7, 36.7, 26.3, 25.7, 21.5 ppm.

IR (**KBr**): 3426, 3026, 2978, 2941, 2314, 2157, 1984, 1865, 1720, 1603, 1501, 1436, 1377, 1262, 1207, 1177, 1058, 996, 923, 820, 789, 704 cm⁻¹.

HRMS (ESI): mass found: 359.16199, mass calculated for $C_{22}H_{24}NaO_3^+$: 359.16177.

methyl 2-(2-hydroxypropan-2-yl)-1-phenyl-3-(p-tolyl)cycloprop-2-ene-1-carboxylate



methyl 1-(4-ethoxyphenyl)-2-(2-hydroxypropan-2-yl)-3-(p-tolyl)cycloprop-2-ene-1carboxylate (3b) ¹**H NMR** (400 MHz, CDCl₃)



methyl 1-(4-fluorophenyl)-2-(2-hydroxypropan-2-yl)-3-(p-tolyl)cycloprop-2-ene-1carboxylate (3c)





methyl 1-(4-chlorophenyl)-2-(2-hydroxypropan-2-yl)-3-(p-tolyl)cycloprop-2-ene-1carboxylate (3d) ¹H NMR (400 MHz, CDCl₃)



methyl 1-(4-bromophenyl)-2-(2-hydroxypropan-2-yl)-3-(p-tolyl)cycloprop-2-ene-1carboxylate (3e) ¹**H NMR** (400 MHz, CDCl₃)



¹³C NMR (101 MHz, CDCl₃)



methyl 1-(3-chlorophenyl)-2-(2-hydroxypropan-2-yl)-3-(p-tolyl)cycloprop-2-ene-1carboxylate (3f) ¹H NMR (600 MHz, CDCl₃)



¹³C NMR (151 MHz, CDCl₃)



methyl 2-(2-hydroxypropan-2-yl)-1-(3-methoxyphenyl)-3-(p-tolyl)cycloprop-2-ene-1carboxylate (3g) ¹**H NMR** (600 MHz, CDCl₃)



¹³C NMR (151 MHz, CDCl₃)



methyl 1-(2-fluorophenyl)-2-(2-hydroxypropan-2-yl)-3-(p-tolyl)cycloprop-2-ene-1carboxylate (3h) ¹**H NMR** (400 MHz, CDCl₃)



¹³C NMR (101 MHz, CDCl₃)





methyl 2-(2-hydroxypropan-2-yl)-1-(naphthalen-2-yl)-3-(p-tolyl)cycloprop-2-ene-1carboxylate (3i) ¹**H NMR** (400 MHz, CDCl₃)



¹³C NMR (101 MHz, CDCl₃)



methyl 2-(2-hydroxypropan-2-yl)-1-(naphthalen-1-yl)-3-(p-tolyl)cycloprop-2-ene-1carboxylate (3j)



2-(2-hydroxypropan-2-yl)-1-phenyl-3-(p-tolyl)cycloprop-2-ene-1-carboxylate ethyl





methyl 2-(2-hydroxypropan-2-yl)-1,3-diphenylcycloprop-2-ene-1-carboxylate (31) ¹H NMR (600 MHz, CDCl₃)

methyl 2-(4-ethylphenyl)-3-(2-hydroxypropan-2-yl)-1-phenylcycloprop-2-ene-1carboxylate (3m)



methyl 2-(4-(tert-butyl)phenyl)-3-(2-hydroxypropan-2-yl)-1-phenylcycloprop-2-ene-1carboxylate (3n) ¹H NMR (400 MHz, CDCl₃)



methyl 2-(4-fluorophenyl)-3-(2-hydroxypropan-2-yl)-1-phenylcycloprop-2-ene-1carboxylate (30) ¹**H NMR** (400 MHz, CDCl₃)



¹³C NMR (101 MHz, CDCl₃)



¹⁹F NMR (376 MHz, CDCl₃)



methyl 2-(2-hydroxypropan-2-yl)-3-(4-iodophenyl)-1-phenylcycloprop-2-ene-1carboxylate (3p) ¹**H NMR** (600 MHz CDCL)



¹H NMR (600 MHz, CDCl₃) -180 -170 -160 -150 -140 ∭ -130 0 -120 `o^{_CH}₃ -110 OH -100 н₃с сн₃ -90 -80 -70 -60 -50 -40 -30 -20 -10 -0 3.00-1 2.98-1 3.05-1 5:03 2:00 1.07 --10 8.5 8.0 7.5 7.0 0 6.5 6.0 5.5 5.0 4.5 f1 (ppm) 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ¹³C NMR (151 MHz, CDCl₃) 107.04 ---52.38 -37.16 29.75 29.71 28.82 -175.45 -500 -450 -400 -350 0 . o_CH₃ -300 OH н₃с сн₃ -250 -200 -150 -100 -50 -0 --50 50 10 60 10 110 100 f1 (ppm) 30 ō 190 180 170 160 150 140 130 120 90 80 70 40 20

methyl 2-([1,1'-biphenyl]-4-yl)-3-(2-hydroxypropan-2-yl)-1-phenylcycloprop-2-ene-1carboxylate (3q)

methyl 2-(4-cyanophenyl)-3-(2-hydroxypropan-2-yl)-1-phenylcycloprop-2-ene-1carboxylate (3r)



methyl 2-(4-acetylphenyl)-3-(2-hydroxypropan-2-yl)-1-phenylcycloprop-2-ene-1carboxylate (3s) ¹**H NMR** (400 MHz, CDCl₃)



methyl 2-(2-hydroxypropan-2-yl)-1-phenyl-3-(4-(trifluoromethoxy)phenyl)cycloprop-2-ene-1-carboxylate (3t) ¹**H NMR** (600 MHz CDCh)



¹⁹F NMR (376 MHz, CDCl₃)



methyl 2-(2-hydroxypropan-2-yl)-1-phenyl-3-(4-(trifluoromethyl)phenyl)cycloprop-2ene-1-carboxylate (3u) ¹H NMR (400 MHz, CDCl₃)





methyl 2-(2-hydroxypropan-2-yl)-3-(3-methoxyphenyl)-1-phenylcycloprop-2-ene-1carboxylate (3v) ¹H NMR (600 MHz, CDCl₃)



methyl 2-(2-hydroxypropan-2-yl)-1-phenyl-3-(m-tolyl)cycloprop-2-ene-1-carboxylate



methyl 2-(2-fluorophenyl)-3-(2-hydroxypropan-2-yl)-1-phenylcycloprop-2-ene-1carboxylate (3x)



¹⁹F NMR (564 MHz, CDCl₃)



methyl 2-(2-hydroxypropan-2-yl)-1-phenyl-3-(o-tolyl)cycloprop-2-ene-1-carboxylate (3y) ¹**H NMR** (400 MHz, CDCl₃)



methyl 2-(2-hydroxypropan-2-yl)-3-(2-methoxyphenyl)-1-phenylcycloprop-2-ene-1carboxylate (3z) ¹H NMR (400 MHz, CDCl₃)



methyl 2-(2-hydroxypropan-2-yl)-1-phenyl-3-(thiophen-2-yl)cycloprop-2-ene-1carboxylate (3aa)



methyl 2-(3,5-bis(trifluoromethyl)phenyl)-3-(2-hydroxypropan-2-yl)-1-phenylcycloprop-2-ene-1-carboxylate (3ab) **H NMR** (400 MHz, CDCl₃)







2-(1-hydroxycyclobutyl)-1-phenyl-3-(p-tolyl)cycloprop-2-ene-1-carboxylate methyl



methyl 2-(1-hydroxycyclopentyl)-1-phenyl-3-(p-tolyl)cycloprop-2-ene-1-carboxylate



2-(1-hydroxycyclohexyl)-1-phenyl-3-(p-tolyl)cycloprop-2-ene-1-carboxylate methyl



methyl 2-(1-hydroxy-1-phenylethyl)-1-phenyl-3-(p-tolyl)cycloprop-2-ene-1carboxylate (3af)





methyl 2-(1-hydroxyethyl)-1,3-diphenylcycloprop-2-ene-1-carboxylate (3ag) ¹H NMR (400 MHz, CDCl₃)



methyl 2-phenyl-2-((3-(p-tolyl)prop-2-yn-1-yl)oxy)acetate (4) ¹H NMR (600 MHz, CDCl₃)

methyl 2-(2-methoxypropan-2-yl)-1-phenyl-3-(p-tolyl)cycloprop-2-ene-1-carboxylate (5) ¹**H NMR** (600 MHz, CDCl₃)

