Supporting Information

for

Visible-light-induced nickel-catalyzed α-hydroxytrifluoroethylolation of alkyl carboxylic acids: Access to trifluoromethyl alkyl acyloins

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Experimental procedures, product characterization, and copies of NMR spectra
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1. General information

$^1$H, $^{19}$F NMR spectra and $^{13}$C NMR spectra were recorded on an Agilent AM400 spectrometer and Bruker AM400 spectrometer. $^1$H NMR and $^{13}$C NMR chemical shifts were reported in ppm relative to chloroform ($^1$H, δ 7.26; $^{13}$C, δ 77.0) or acetone ($^1$H, δ 2.05; $^{13}$C, δ 29.8) and $^{19}$F NMR chemical shifts were determined relative to CFCl$_3$ as internal standard. Chemical shifts (δ) were reported in ppm and coupling constants ($J$) are in Hertz (Hz). The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. IR spectra were recorded on a Thermo Scientific Nicolet iS5 Fourier transform infrared (FT-IR) spectrometer and were reported in terms of wavenumber of absorption (cm$^{-1}$). Detection of melting point was conducted on the SGW X-4A microscopic melting point meter. High-resolution mass spectrometry (HRMS) was performed on a Waters Premier GC-TOF MS instrument with electron impact (EI) mode, on a JEOL-AccuTOF-GCv4G-GCT MS instrument with field ionization (FI) mode or on a Thermo Scientific Q Exactive HF Orbitrap-FTMS instrument with electrospray ionization (ESI) mode.

All photochemical reactions were performed in 4 mL screw cap scintillation vials (unless noted otherwise) under an inert atmosphere at room temperature and irradiated with two 20 W purple LEDs (maximum emission wavelength = 399 nm).

![Figure S1: The emission spectra of the purple LEDs.](image-url)
2. Preparation of substrates

Carboxylic acid substrates and the nickel catalysts (NiBr$_2$(dtbbpy), CAS: 1894189-67-3; NiBr$_2$(bpv), CAS: 46389-47-3; NiBr$_2$(Phen), CAS: 48165-50-0; NiCl$_2$(dtbbpy), CAS: 1034901-50-2) were obtained commercially and used as received. And the N-alkoxyphthalimide reagent 2 (2-(2,2,2-trifluoroethoxy)isoindoline-1,3-dione)$^1$ were synthesized according to the literature procedures.

3. General procedures and substrate scope

In the glove box with nitrogen atmosphere, to a 8 mL vial equipped with a magnetic stir bar, NiBr$_2$(dtbbpy) (19.6 mg, 0.04 mmol, 10 mol %), alkyl carboxylic acid 1(0.4 mmol, 1.0 equiv), 2 (147.1 mg, 0.6 mmol, 1.5 equiv), Hantzsch ester (152.0 mg, 0.6 mmol, 1.5 equiv), and 4.0 mL of anhydrous N,N-dimethylacetamide were added. The vial was then re-capped and taken out of the glove box, and Piv$_2$O (111.8 mg, 0.6 mmol, 1.5 equiv), H$_2$O (21.6 mg, 1.2 mmol, 3.0 equiv) were added. The vial was sealed with parafilm and the reaction mixture was then stirred under irradiation by purple LEDs ($\lambda_{max} = 399$ nm) for 7 h. After the reaction was complete, the reaction mixture was poured into water and extracted with EtOAc. The combined organic phase was separated and washed with brine, dried over Na$_2$SO$_4$, and concentrated under vacuum. The resulting residue was purified by silica gel flash column chromatography to give the product.

1,1,1-Trifluoro-2-hydroxy-6-phenylhexan-3-one (3a)

The residue was purified by silica gel column chromatography (hexane/acetone = 15:1) to afford 3a (69.9 mg, 71%) as a white solid. M.P. 90.7-91.3 $^\circ$C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.33-7.28 (m, 2H), 7.24-7.20 (m, 1H), 7.19-7.15 (m, 2H), 4.49-4.41 (m, 1H), 4.02 (s, 1H), 2.79-2.56 (m, 4H), 2.09-1.99 (m, 2H). $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ -74.14 (d, $J = 7.6$ Hz, 3F). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 202.7, 140.7, 128.6, 128.4, 126.3, 122.4 (q, $J = 283.8$ Hz), 74.8 (q, $J = 31.7$ Hz), 38.8 (q, $J = 2.2$ Hz), 34.7, 24.7. IR (thin film): $\nu$ 3405, 2955, 1725, 1467, 1270, 1142, 1118, 764, 705 cm$^{-1}$. MS (EI): $m/z$ 246.1 [M$^+$]; HRMS (EI-TOF) $m/z$: [M$^+$] Calcd. for C$_{12}$H$_{13}$F$_3$O$_2$ 246.0862; Found: 246.0866.
1,1,1-Trifluoro-2-hydroxy-6-(4-methoxyphenyl)hexan-3-one (3b)

\[
\text{MeO} \quad \text{CF}_3 \quad \text{OH}
\]

The residue was purified by silica gel column chromatography (hexane/acetone = 15:1) to afford 3b (71.8 mg, 65%) as a white solid. M.P. 65.5-66.7 °C. \( ^1H \) NMR (400 MHz, CDCl\(_3\)) \( \delta \) 7.07 (d, \( J = 8.5 \) Hz, 2H), 6.84 (d, \( J = 8.6 \) Hz, 2H), 4.44 (q, \( J = 7.6 \) Hz, 1H), 4.01 (s, 1H), 3.79 (s, 3H), 2.75-2.54 (m, 4H), 2.06-1.94 (m, 2H). \( ^19F \) NMR (376 MHz, CDCl\(_3\)) \( \delta \) -74.14 (d, \( J = 7.5 \) Hz, 3F). \( ^{13}C \) NMR (101 MHz, CDCl\(_3\)) \( \delta \) 202.7, 158.0, 132.7, 129.3, 122.4 (q, \( J = 283.1 \) Hz), 113.9, 74.8 (q, \( J = 31.7 \) Hz), 55.2, 38.7 (q, \( J = 2.4 \) Hz), 33.8, 24.9. IR (thin film): \( \nu \) 3438, 2941, 1725, 1614, 1515, 1342, 1249, 1146, 1006, 676, 557 cm\(^{-1}\). MS (EI): \( m/z \) 276.1 [M\(^+\)]; HRMS (EI-TOF) \( m/z \): [M\(^+\)] Calcd. for C\(_{13}\)H\(_{15}\)F\(_3\)O\(_2\) 276.0968; Found: 276.0967.

1,1,1-Trifluoro-2-hydroxy-6-(p-tolyl)hexan-3-one (3c)

\[
\text{Me} \quad \text{CF}_3 \quad \text{OH}
\]

The residue was purified by silica gel column chromatography (hexane/acetone = 15:1) to afford 3c (64.5 mg, 62%) as a white solid. M.P. 60.7-61.5 °C. \( ^1H \) NMR (400 MHz, CDCl\(_3\)) \( \delta \) 7.14-7.04 (m, 4H), 4.45 (q, \( J = 7.6 \) Hz, 1H), 3.86 (s, 1H), 2.78-2.56 (m, 4H), 2.33 (s, 3H), 2.06-1.96 (m, 2H). \( ^19F \) NMR (376 MHz, CDCl\(_3\)) \( \delta \) -74.14 (d, \( J = 7.5 \) Hz, 3F). \( ^{13}C \) NMR (101 MHz, CDCl\(_3\)) \( \delta \) 202.7, 137.6, 135.7, 129.2, 128.2, 122.4 (q, \( J = 283.1 \) Hz), 74.8 (q, \( J = 31.7 \) Hz), 38.8 (q, \( J = 2.4 \) Hz), 34.2, 24.8, 21.0. IR (thin film): \( \nu \) 3416, 2925, 1725, 1516, 1458, 1346, 1249, 1080, 805, 676 cm\(^{-1}\). MS (EI): \( m/z \) 260.1 [M\(^+\)]; HRMS (EI-TOF) \( m/z \): [M\(^+\)] Calcd. for C\(_{13}\)H\(_{15}\)F\(_3\)O\(_2\) 260.1019; Found: 260.1023.

6-(4-Chlorophenyl)-1,1,1-trifluoro-2-hydroxyhexan-3-one (3d)

\[
\text{Cl} \quad \text{CF}_3 \quad \text{OH}
\]

The residue was purified by silica gel column chromatography (hexane/acetone = 15:1) to afford 3d (44.9 mg, 40%) as a white solid. M.P. 75.4-77.0 °C. \( ^1H \) NMR (400 MHz, CDCl\(_3\)) \( \delta \) 7.25 (d, \( J = 8.3 \) Hz, 2H), 7.08 (d, \( J = 8.3 \) Hz, 2H), 4.45 (q, \( J = 7.5 \) Hz, 1H), 3.84 (s, 1H), 2.75-2.53 (m, 4H), 2.06-1.91 (m, 2H). \( ^19F \) NMR (376 MHz, CDCl\(_3\)) \( \delta \) -74.14 (d, \( J = 7.5 \) Hz, 3F). \( ^{13}C \) NMR (101 MHz, CDCl\(_3\)) \( \delta \) 202.5, 139.2, 132.0, 129.7, 128.6, 122.4 (q, \( J = 283.3 \) Hz), 74.8 (q, \( J = 31.5 \) Hz), 38.7 (q, \( J = 2.3 \) Hz), 34.0, 24.5. IR (thin film): \( \nu \) 3416, 2951, 1727, 1493, 1460, 1407, 1269, 1142, 1004, 806, 527 cm\(^{-1}\).
1. MS (EI): m/z 280.0 [M⁺]; HRMS (EI-TOF) m/z: [M⁺] Calcd. for C_{12}H_{12}ClF_3O_2 280.0472; Found: 280.0476.

6-(4-Bromophenyl)-1,1,1-trifluoro-2-hydroxyhexan-3-one (3e)

The residue was purified by silica gel column chromatography (hexane/acetone = 15:1) to afford 3e (71.5 mg, 55%) as a white solid. M.P. 91.2-92.7 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.41 (d, J = 8.3 Hz, 2H), 7.04 (d, J = 8.3 Hz, 2H), 4.46 (q, J = 7.5 Hz, 1H), 4.00 (s, 1H), 2.78-2.55 (m, 4H), 2.05-1.94 (m, 2H). ¹⁹F NMR (376 MHz, CDCl₃) δ -74.12 (d, J = 7.5 Hz, 3F). ¹³C NMR (101 MHz, CDCl₃) δ 202.5, 139.7, 131.6, 130.1, 122.4 (q, J = 283.2 Hz), 120.0, 74.8 (q, J = 31.7 Hz), 38.7 (q, J = 2.4 Hz), 34.1, 24.4. IR (thin film): ν 3422, 2950, 1724, 1489, 1341, 1259, 1144, 1010, 836, 677 cm⁻¹. MS (EI): m/z 324.0 [M⁺]; HRMS (EI-TOF) m/z: [M⁺] Calcd. for C_{12}H_{12}BrF_3O_2 323.9967; Found: 323.9963.

1,1,1-Trifluoro-5-(4-fluorophenyl)-2-hydroxypentan-3-one (3f)

The residue was purified by silica gel column chromatography (hexane/acetone = 15:1) to afford 3f (68.1 mg, 68%) as a sticky yellowish oil. ¹H NMR (400 MHz, CDCl₃) δ 7.16-7.12 (m, 2H), 7.02-6.95 (m, 2H), 4.45 (q, J = 7.5 Hz, 1H), 4.02 (s, 1H), 3.08-2.87 (m, 4H). ¹⁹F NMR (376 MHz, CDCl₃) δ -74.12 (d, J = 7.5 Hz, 3F), -116.41 – -116.51 (m, 1F). ¹³C NMR (101 MHz, CDCl₃) δ 201.9, 161.6 (d, J = 244.8 Hz), 135.2, 129.7 (d, J = 8.0 Hz), 122.4 (q, J = 283.3 Hz), 115.5 (d, J = 21.3 Hz), 75.0 (q, J = 31.6 Hz), 41.4, 28.4. IR (thin film): ν 3454, 2929, 1731, 1489, 1341, 1259, 1144, 1010, 836, 677 cm⁻¹. MS (EI): m/z 250.1 [M⁺]; HRMS (EI-TOF) m/z: [M⁺] Calcd. for C_{11}H_{10}F_4O_2 250.0611; Found: 250.0609.

1,1,1-Trifluoro-5-(3-fluorophenyl)-2-hydroxypentan-3-one (3g)

The residue was purified by silica gel column chromatography (hexane/acetone = 15:1) to afford 3g (70.1 mg, 70%) as a sticky yellowish oil. ¹H NMR (400 MHz, CDCl₃) δ 7.29-7.23 (m, 1H), 6.98-6.87 (m, 3H), 4.53-4.41 (m, 1H), 3.99 (d, J = 6.3 Hz, 1H), 3.09-2.91 (m, 4H). ¹⁹F NMR (376 MHz, CDCl₃) δ -74.08 (d, J = 7.5 Hz, 3F), -113.00
-113.08 (m, 1F). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 201.7, 162.9 (d, $J = 246.1$ Hz), 142.0 (d, $J = 7.3$ Hz), 130.2 (d, $J = 8.3$ Hz), 123.9 (d, $J = 3.0$ Hz), 122.3 (q, $J = 283.2$ Hz), 115.2 (d, $J = 21.1$ Hz), 113.5 (d, $J = 20.9$ Hz), 75.0 (q, $J = 31.7$ Hz), 40.9 (q, $J = 2.3$ Hz), 28.8.

IR (thin film): ν 3448, 2926, 2855, 1730, 1618, 1491, 1190, 1119, 782, 520 cm$^{-1}$. MS (EI): $m/\ell$ 250.1 [M$^+$]; HRMS (EI-TOF) $m/\ell$: [M$^+$] Calcd. for C$_{11}$H$_{10}$F$_4$O$_2$ 250.0611; Found: 250.0614.

1,1,1-Trifluoro-2-hydroxy-5-phenylpentan-3-one (3h)

![3h](image)

The residue was purified by silica gel column chromatography (hexane/acetone = 15:1) to afford 3h (65.0 mg, 70%) as a sticky yellowish oil. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.34-7.29 (m, 2H), 7.26-7.16 (m, 3H), 4.51-4.39 (m, 1H), 4.02 (d, $J = 6.2$ Hz, 1H), 3.09-2.92 (m, 4H). $^{19}$F NMR (376 MHz, CDCl$_3$) δ -74.04 (d, $J = 7.5$ Hz, 3F). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 202.1, 139.5, 128.7, 128.2, 126.6, 122.4 (q, $J = 283.2$ Hz), 75.0 (q, $J = 31.7$ Hz), 41.3 (q, $J = 2.4$ Hz), 29.3. IR (thin film): ν 3448, 3030, 2928, 1729, 1497, 1250, 1189, 1119, 751, 700 cm$^{-1}$. MS (EI): $m/\ell$ 232.1 [M$^+$]; HRMS (EI-TOF) $m/\ell$: [M$^+$] Calcd. for C$_{11}$H$_{11}$F$_3$O$_2$ 232.0706; Found: 232.0709.

6-(4-Chlorophenoxy)-1,1,1-trifluoro-2-hydroxyhexan-3-one (3i)

![3i](image)

The residue was purified by silica gel column chromatography (hexane/acetone = 15:1–10:1) to afford 3i (73.6 mg, 62%) as a sticky yellowish oil. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.25-7.20 (m, 2H), 6.82-6.76 (m, 2H), 4.62-4.54 (m, 1H), 4.04 (d, $J = 6.3$ Hz, 1H), 4.00-3.93 (m, 2H), 2.96-2.81 (m, 2H), 2.24-2.16 (m, 2H). $^{19}$F NMR (376 MHz, CDCl$_3$) δ -74.16 (d, $J = 7.8$ Hz, 3F). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 202.4, 139.5, 129.4, 125.9, 122.4 (q, $J = 283.3$ Hz), 115.6, 74.8 (q, $J = 31.6$ Hz), 66.5, 36.4 (q, $J = 2.4$ Hz), 23.3. IR (thin film): ν 3454, 2919, 1729, 1597, 1494, 1244, 1093, 826, 663 cm$^{-1}$. MS (EI): $m/\ell$ 296.0 [M$^+$]; HRMS (EI-TOF) $m/\ell$: [M$^+$] Calcd. for C$_{12}$H$_{12}$ClF$_3$O$_3$ 296.0422; Found: 296.0421.

1,1,1-Trifluoro-5-(furan-2-y1)-2-hydroxypentan-3-one (3j)

![3j](image)
The residue was purified by silica gel column chromatography (hexane/acetonitrile = 15:1) to afford 3j (53.3 mg, 60%) as a sticky yellowish oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.33-7.27 (m, 1H), 6.29-6.27 (m, 1H), 6.05-6.02 (m, 1H), 4.57-4.45 (m, 1H), 4.02 (d, $J$ = 6.2 Hz, 1H), 3.09-2.97 (m, 4H). $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ -74.16 (d, $J$ = 7.5 Hz, 3F). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 201.7, 152.9, 141.5, 122.4 (q, $J$ = 283.2 Hz), 110.3, 105.8, 74.9 (q, $J$ = 31.7 Hz), 38.1 (q, $J$ = 2.4 Hz), 21.8. IR (thin film): $\nu$ 3406, 2959, 2926, 1731, 1508, 1407, 1266, 1000, 741, 598 cm$^{-1}$. MS (EI): m/z 222.1 [M$^+$]; HRMS (EI-TOF) m/z: [M$^+$] Calcd. for C$_9$H$_9$F$_3$O$_3$ 222.0498; Found: 222.0501.

1,1,1-Trifluoro-2-hydroxy-5-(thiophen-2-yl)pentan-3-one (3k)

The residue was purified by silica gel column chromatography (hexane/acetonitrile = 15:1) to afford 3k (55.3 mg, 58%) as a sticky yellowish oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.17-7.13 (m, 1H), 6.96-6.90 (m, 1H), 6.86-6.78 (m, 1H), 4.54-4.42 (m, 1H), 4.02 (d, $J$ = 5.5 Hz, 1H), 3.27-3.21 (m, 2H), 3.15-2.96 (m, 2H). $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ -74.07 (d, $J$ = 7.5 Hz, 3F). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 201.6, 141.9, 127.0, 125.1, 123.8, 122.3 (q, $J$ = 283.3 Hz), 75.1 (q, $J$ = 31.7 Hz), 41.5 (q, $J$ = 2.3 Hz), 23.4. IR (thin film): $\nu$ 3455, 2926, 2856, 1731, 1441, 1250, 1116, 996, 700 cm$^{-1}$. MS (EI): m/z 238.0 [M$^+$]; HRMS (EI-TOF) m/z: [M$^+$] Calcd. for C$_9$H$_9$F$_3$O$_2$ 238.0270; Found: 238.0271.

5-(Benzo[d][1,3]dioxol-5-yl)-1,1,1-trifluoro-2-hydroxypentan-3-one (3l)

The residue was purified by silica gel column chromatography (hexane/acetonitrile = 15:1) to afford 3l (42.0 mg, 38%) as a sticky yellowish oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 6.75-6.61 (m, 3H), 5.93 (s, 2H), 4.50-4.40 (m, 1H), 4.04 (d, $J$ = 6.2 Hz, 1H), 3.03-2.87 (m, 4H). $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ -74.04 (d, $J$ = 7.5 Hz, 3F). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 202.1, 147.8, 146.2, 133.3, 122.4 (q, $J$ = 283.3 Hz), 121.1, 108.7, 108.4, 100.9, 75.0 (q, $J$ = 31.6 Hz), 41.6 (q, $J$ = 2.2 Hz), 29.0. IR (thin film): $\nu$ 3444, 2905, 1729, 1505, 1493, 1247, 1189, 1117, 1040, 931, 810 cm$^{-1}$. MS (EI): m/z 276.1 [M$^+$]; HRMS (EI-TOF) m/z: [M$^+$] Calcd. for C$_{12}$H$_{11}$F$_3$O$_4$ 276.0604; Found: 276.0606.
The residue was purified by silica gel column chromatography (hexane/acetone = 15:1) to afford 3m (51.8 mg, 42%) as a white solid. **M.P.** 148.4-149.0 °C. **1H NMR** (400 MHz, (CD$_3$)$_2$CO) δ 7.87-7.82 (m, 4H), 6.19 (d, $J = 6.7$ Hz, 1H), 4.79 (q, $J = 7.9$ Hz, 1H), 3.95 (t, $J = 7.2$ Hz, 2H), 3.29-3.14 (m, 2H). **19F NMR** (376 MHz, (CD$_3$)$_2$CO) δ -75.44 (d, $J = 8.0$ Hz, 3F). **13C NMR** (101 MHz, (CD$_3$)$_2$CO) δ 203.3, 168.5, 135.0, 133.1, 124.3 (q, $J = 282.8$ Hz), 123.7, 75.2 (q, $J = 29.6$ Hz), 38.2 (q, $J = 1.9$ Hz), 33.2. **IR (thin film):** ν 3434, 2924, 1770, 1709, 1244, 1180, 992, 719, 529 cm$^{-1}$. **MS (EI):** m/z 301.1 [M$^+$]; **HRMS (EI-TOF) m/z:** [M$^+$] Calcd. for C$_{13}$H$_{10}$F$_3$NO$_4$ 301.0556; Found: 301.0548.

The residue was purified by silica gel column chromatography (hexane/acetone = 15:1) to afford 3n (53.7 mg, 55%) as a sticky yellowish oil. Product was isolated as a 1:0.8 mixture of two isomers. **1H NMR** (400 MHz, CDCl$_3$) δ 7.36-7.23 (m, 3H), 7.17-7.11 (m, 2H), 4.77-4.67 (m, 1H), 4.18-4.10 (m, 1H), 2.86-2.73 (m, 1H), 2.47-2.39 (m, 1H), 2.00-1.89 (m, 1H), 1.77-1.67 (m, 1H). **19F NMR** (376 MHz, CDCl$_3$) δ -73.83 (d, $J = 7.5$ Hz, 1.67F), -73.98 (d, $J = 7.4$ Hz, 1.33F). **13C NMR** (101 MHz, CDCl$_3$) δ 201.1, 201.0, 138.5, 138.3, 128.7, 128.6, 127.2, 126.6, 126.1, 122.6 (q, $J = 283.5$ Hz), 122.5 (q, $J = 283.3$ Hz), 75.2 (q, $J = 31.5$ Hz), 32.8, 32.1, 30.3 (q, $J = 3.2$ Hz), 29.8 (q, $J = 2.8$ Hz), 21.3, 20.0. **IR (thin film):** ν 3447, 3032, 2929, 1770, 1709, 1244, 1180, 992, 719, 529 cm$^{-1}$. **MS (EI):** m/z 244.1 [M$^+$]; **HRMS (EI-TOF) m/z:** [M$^+$] Calcd. for C$_{12}$H$_{11}$F$_3$O$_2$ 244.0706; Found: 244.0705.

The residue was purified by silica gel column chromatography (hexane/acetone = 15:1) to afford 3o (76.4 mg, 74%) as a sticky yellowish oil. Product was isolated as a 7/2 mixture of two isomers. **1H NMR** (400 MHz, CDCl$_3$) δ 7.38-7.31 (m, 2H), 7.27-7.21

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(m, 3H), 4.65-4.52 (m, 1H), 4.16-4.07 (m, 1H), 3.78-3.52 (m, 2H), 2.85-2.63 (m, 2H), 2.57-2.43 (m, 2H). \(^{19}F\) NMR (376 MHz, CDCl\textsubscript{3}) \(\delta -73.99\) (d, \(J = 7.6\) Hz, 0.67F), \(-74.04\) (d, \(J = 7.6\) Hz, 2.33F). \(^{13}C\) NMR (101 MHz, CDCl\textsubscript{3}) \(\delta 204.7, 203.5, 144.3, 143.7, 128.5, 128.4, 126.5, 126.4, 126.3, 126.2, 122.51\) (q, \(J = 283.0\) Hz), \(122.48\) (q, \(J = 283.1\) Hz), 73.7 (q, \(J = 31.8\) Hz), 73.6 (q, \(J = 31.8\) Hz), 39.0 (q, \(J = 2.2\) Hz), 38.3 (q, \(J = 2.3\) Hz), 36.0, 35.2, 32.7, 32.5, 31.6, 31.0. IR (thin film): \(\nu 3450, 2988, 2945, 1718, 1603, 1348, 1248, 1156, 755\) cm\(^{-1}\). MS (EI): \(m/z\) 258.1 [M\(^+\)]; HRMS (EI-TOF) \(m/z\): [M\(^+\)] Calcd. for C\textsubscript{13}H\textsubscript{13}F\textsubscript{3}O\textsubscript{2} 258.0862; Found: 258.0860.

1-(3-(Benzyloxy)cyclobutyl)-3,3,3-trifluoro-2-hydroxypropan-1-one (3p)

![3p](image)

The residue was purified by silica gel column chromatography (hexane/acetone = 15:1) to afford 3p (86.5 mg, 75%) as a sticky yellowish oil. Product was isolated as a 2/1 mixture of two isomers. \(^{1}H\) NMR (400 MHz, CDCl\textsubscript{3}) \(\delta 7.39-7.27\) (m, 5H), 4.57-4.47 (m, 1H), 4.44-4.39 (m, 2H), 4.31-3.99 (m, 2H), 3.62-2.98 (m, 1H), 2.61-2.45 (m, 2H), 2.43-2.23 (m, 2H). \(^{19}F\) NMR (376 MHz, CDCl\textsubscript{3}) \(\delta -73.96\) (d, \(J = 7.8\) Hz, 1F), \(-74.10\) (d, \(J = 7.6\) Hz, 2F). \(^{13}C\) NMR (101 MHz, CDCl\textsubscript{3}) \(\delta 205.0, 202.9, 137.73, 137.66, 128.43, 128.42, 128.02, 127.80, 122.4\) (q, \(J = 283.1\) Hz), 73.94 (q, \(J = 31.8\) Hz), 73.79 (q, \(J = 31.7\) Hz), 70.6, 70.4, 70.2, 67.9, 36.2 (q, \(J = 2.3\) Hz), 33.90, 33.86, 33.6 (q, \(J = 2.2\) Hz), 33.0, 32.6. IR (thin film): \(\nu 3441, 3032, 2945, 1722, 1496, 1455, 1357, 1248, 1127, 740, 699\) cm\(^{-1}\). MS (EI): \(m/z\) 288.1 [M\(^+\)]; HRMS (EI-TOF) \(m/z\): [M\(^+\)] Calcd. for C\textsubscript{14}H\textsubscript{15}F\textsubscript{3}O\textsubscript{3} 288.0968; Found: 288.0965.

1-(2,3-Dihydro-1H-inden-2-yl)-3,3,3-trifluoro-2-hydroxypropan-1-one (3q)

![3q](image)

The residue was purified by silica gel column chromatography (hexane/acetone = 15:1) to afford 3q (63.5 mg, 65%) as a white solid. M.P. 60.9-61.7 °C. \(^{1}H\) NMR (400 MHz, CDCl\textsubscript{3}) \(\delta 7.26-7.18\) (m, 4H), 4.73 (q, \(J = 7.7\) Hz, 1H), 4.07 (s, 1H), 3.85-3.76 (m, 1H), 3.44-3.34 (m, 2H), 3.20-3.12 (m, 2H). \(^{19}F\) NMR (376 MHz, CDCl\textsubscript{3}) \(\delta -73.78\) (d, \(J = 7.5\) Hz, 3F). \(^{13}C\) NMR (101 MHz, CDCl\textsubscript{3}) \(\delta 204.5, 140.9, 139.7, 127.2, 126.9, 124.5, 124.3, 122.5\) (q, \(J = 283.4\) Hz), 74.1 (q, \(J = 31.5\) Hz), 48.0 (q, \(J = 2.1\) Hz), 36.7, 34.8. IR (thin film): \(\nu 3408, 3048, 2930, 2854, 1723, 1488, 1326, 1261, 1124, 1002, 758, 528\) cm\(^{-1}\). MS (EI): \(m/z\) 244.1 [M\(^+\)]; HRMS (EI-TOF) \(m/z\): [M\(^+\)] Calcd. for C\textsubscript{12}H\textsubscript{11}F\textsubscript{3}O\textsubscript{2} 244.0706; Found: 244.0703.
3,3,3-Trifluoro-2-hydroxy-1-(1,2,3,4-tetrahydronaphthalen-2-yl)propan-1-one (3r)

\[
\text{\textbf{OH} \quad \text{CF}_3}
\]

The residue was purified by silica gel column chromatography (hexane/acetone = 15:1) to afford 3r (56.8 mg, 55%) as a sticky yellowish oil. Product was isolated as a 1:1 mixture of two isomers. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta 7.20-7.08 \text{ (m, 4H), 4.84-4.71 (m, 1H), 4.12-4.08 (m, 1H), 3.24-2.84 (m, 5H), 2.25-2.12 (m, 1H), 1.97-1.77 (m, 1H).}\) \(^{19}\)F NMR (376 MHz, CDCl\(_3\)) \(\delta -73.74 \text{ (d, } J = 7.5 \text{ Hz, 1.5F), -73.77 (d, } J = 7.6 \text{ Hz, 1.5F).}\) \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta 205.7, 205.6, 135.5, 134.7, 134.1, 133.5, 129.1, 129.04, 128.85, 128.81, 126.5, 126.21, 126.20, 126.1, 122.55 (q, } J = 283.3 \text{ Hz), 122.52 (q, } J = 283.6 \text{ Hz), 73.74 (q, } J = 31.5 \text{ Hz), 73.24 (q, } J = 31.4 \text{ Hz), 44.4-44.2 (m), 32.4, 30.0, 28.6, 28.0, 26.5, 24.4. IR (thin film): } \nu 3454, 3063, 2932, 1722, 1497, 1454, 1247, 1180, 747, 434 \text{ cm}^{-1}. MS (EI): } m/z 258.1 [M^+]; HRMS (EI-TOF) } m/z: [M^+] Calcd. for C\(_{13}\)H\(_{13}\)F\(_3\)O\(_2\) 258.0862; Found: 258.0868.

1-(4-(4-Chlorophenyl)cyclohexyl)-3,3,3-trifluoro-2-hydroxypropan-1-one (3s)

\[
\text{\textbf{OH} \quad \text{CF}_3}
\]

The residue was purified by silica gel column chromatography (hexane/acetone = 15:1) to afford 3s (89.8 mg, 70%) as a white solid. M.P. 127.7-129.0°C. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta 7.28-7.23 \text{ (m, 2H), 7.14-7.09 (m, 2H), 4.72-4.61 (m, 1H), 4.04-3.99 (m, 1H), 2.84-2.76 (m, 1H), 2.57-2.48 (m, 1H), 2.13-1.94 (m, 4H), 1.77-1.40 (m, 4H).}\) \(^{19}\)F NMR (376 MHz, CDCl\(_3\)) \(\delta -73.72 \text{ (d, } J = 7.5 \text{ Hz, 3F).}\) \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta 206.0, 144.6, 131.9, 128.5, 128.0, 122.5 (q, } J = 283.5 \text{ Hz), 73.3 (q, } J = 31.3 \text{ Hz), 47.0 (q, } J = 2.1 \text{ Hz), 42.7, 33.3, 32.4, 29.8, 27.3. IR (thin film): } \nu 3368, 2942, 2864, 1724, 1497, 1454, 1247, 1180, 747, 434 \text{ cm}^{-1}. MS (EI): } m/z 320.1 [M^+]; HRMS (EI-TOF) } m/z: [M^+] Calcd. for C\(_{15}\)H\(_{16}\)ClF\(_3\)O\(_2\) 320.0785; Found: 320.0783.

6-(4-(Bis(2-chloroethyl)amino)phenyl)-1,1,1-trifluoro-2-hydroxyhexan-3-one (3t)

\[
\text{\textbf{OH} \quad \text{CF}_3}
\]
The residue was purified by silica gel column chromatography (hexane/acetone = 15:1–10:1) to afford \(3t\) (100.4 mg, 65%) as a sticky yellowish oil. \(^1H\) NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.06 (d, \(J = 8.6\) Hz, 2H), 6.64 (d, \(J = 8.7\) Hz, 2H), 4.52-4.40 (m, 1H), 4.03 (d, \(J = 6.3\) Hz, 1H), 3.75-3.68 (m, 4H), 3.67-3.60 (m, 4H), 2.79-2.49 (m, 4H), 2.05-1.93 (m, 2H). \(^{19}F\) NMR (376 MHz, CDCl\(_3\)) \(\delta\) -74.08 (d, \(J = 7.9\) Hz, 3F). \(^{13}C\) NMR (101 MHz, CDCl\(_3\)) \(\delta\) 202.7, 144.5, 129.8, 129.6, 122.4 (q, \(J = 283.3\) Hz), 112.2, 74.8 (q, \(J = 31.6\) Hz), 53.5, 40.5, 38.8 (q, \(J = 2.3\) Hz), 33.5, 24.9. IR (thin film): \(\nu\) 3451, 2930, 1727, 1615, 1519, 1355, 1248, 1181, 1015, 742 cm\(^{-1}\). MS (ESI): \(m/z\) 386.1 [M+H]\(^+\); HRMS (ESI-TOF) \(m/z\): [M+H]\(^+\) Calcd. for C\(_{16}\)H\(_{21}\)Cl\(_2\)F\(_3\)NO\(_2\) 386.0896; Found: 386.0895.

\[
\]

The residue was purified by silica gel column chromatography (hexane/acetone = 10:1–5:1) to afford \(3u\) (135.7 mg, 70%) as a white solid. Product was isolated as a 1:1 mixture of two isomers. M.P. 207.9-208.5°C. \(^1H\) NMR (400 MHz, CDCl\(_3\)) \(\delta\) 4.56-4.44 (m, 1H), 4.19-4.12 (m, 1H), 2.95-2.77 (m, 3H), 2.74-2.53 (m, 2H), 2.38-2.07 (m, 8H), 2.03-1.78 (m, 6H), 1.66-1.53 (m, 1H), 1.48-1.40 (m, 1H), 1.38 (s, 3H), 1.32-1.18 (m, 3H), 1.04 (s, 3H), 0.86-0.77 (m, 3H). \(^{19}F\) NMR (376 MHz, CDCl\(_3\)) \(\delta\) -74.08 (d, \(J = 7.6\) Hz, 1.5F), -74.13 (d, \(J = 7.5\) Hz, 1.5F). \(^{13}C\) NMR (101 MHz, CDCl\(_3\)) \(\delta\) 211.92, 211.89, 209.1, 208.6, 203.3, 203.2, 122.4 (q, \(J = 283.3\) Hz), 74.85 (q, \(J = 31.4\) Hz), 74.58 (q, \(J = 31.3\) Hz), 56.8, 51.70, 51.67, 48.9, 46.7, 45.5, 45.4, 45.3, 44.9, 42.7, 38.5, 37.0-36.7 (m), 36.7-36.4 (m), 36.4, 35.9, 35.3, 35.16, 35.15, 28.7, 28.6, 27.6, 27.5, 25.0, 21.8, 18.6, 18.5, 11.73, 11.71. IR (thin film): \(\nu\) 3446, 2967, 1712, 1468, 1273, 1173, 1125, 912, 733 cm\(^{-1}\). MS (ESI): \(m/z\) 485.3 [M+H]\(^+\); HRMS (ESI-TOF) \(m/z\): [M+H]\(^+\) Calcd. for C\(_{28}\)H\(_{36}\)F\(_3\)O\(_5\) 485.2509; Found: 485.2507.
4. Scale-up reaction

In the glove box with nitrogen atmosphere, to a 25 mL vial equipped with a magnetic stir bar, NiBr$_2$(dtbbpy) (48.9 mg, 0.1 mmol, 10 mol %), 4-phenylbutyric acid 1a (1.0 mmol, 1.0 equiv, 164.2 mg), 2 (367.5 mg, 1.5 mmol, 1.5 equiv), Hantzsch ester (379.9 mg, 1.5 mmol, 1.5 equiv), and 10.0 mL of anhydrous N,N-dimethylacetamide were added, the vial was then re-capped and taken out of the glove box, and Piv$_2$O (279.3 mg, 1.5 mmol, 1.5 equiv), H$_2$O (54.0 mg, 3.0 mmol, 3.0 equiv) were added. The vial was sealed with parafilm and the reaction mixture was then stirred under irradiation by purple LEDs ($\lambda_{\text{max}}$ = 399 nm) for 7 h. After the reaction was complete, the reaction mixture was poured into water and extracted with EtOAc. The combined organic phase was separated and washed with brine, dried over Na$_2$SO$_4$, and concentrated under vacuum. The resulting residue was purified by silica gel flash column chromatography (hexane/acetone = 15:1) to afford 3a (157.4 mg, 64%) as a white solid.
5. ORTEP Drawing of the X-ray crystallographic structure

5.1 1,1,1-Trifluoro-2-hydroxy-6-phenylhexan-3-one (3a)

The crystals were obtained from a solution of chloroform and hexane upon slow volatilization.

The crystal structure has been deposited at the Cambridge Crystallographic Data Center and allocated the deposition numbers CCDC 2222184. The thermal ellipsoids are shown at the 30% probability level. This data can be obtained free of charge from the Cambridge Crystallographic Data Center via www.ccdc.cam.ac.uk/data_request/cif
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5.2 1-(4-(4-Chlorophenyl)cyclohexyl)-3,3,3-trifluoro-2-hydroxypropan-1-one (3s)

The crystals were obtained from a solution of chloroform and hexane upon slow volatilization.

The crystal structure has been deposited at the Cambridge Crystallographic Date Center and allocated the deposition numbers CCDC 2234475. The thermal ellipsoids are shown at the 30% probability level. This data can be obtained free of charge from the Cambridge Crystallographic Date Center via [www.ccdc.cam.ac.uk/data_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif)
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6. References

7. Copies of $^1$H NMR, $^{19}$F NMR and $^{13}$C NMR spectra of products

$^1$H NMR (400 MHz, CDCl$_3$)

$^{19}$F NMR (376 MHz, CDCl$_3$)
$^{13}$C NMR (101 MHz, CDCl$_3$)

![Carbon-13 NMR spectrum for compound 3a](image)

$^1$H NMR (400 MHz, CDCl$_3$)

![Proton NMR spectrum for compound 3b](image)
$^{19}$F NMR (376 MHz, CDCl$_3$)

$^{13}$C NMR (101 MHz, CDCl$_3$)
$^1$H NMR (400 MHz, CDCl$_3$)

$^{19}$F NMR (376 MHz, CDCl$_3$)
$^{13}$C NMR (101 MHz, CDCl$_3$)

$^1$H NMR (400 MHz, CDCl$_3$)
$^{19}$F NMR (376 MHz, CDCl$_3$)

$^{13}$C NMR (101 MHz, CDCl$_3$)
$^1$H NMR (400 MHz, CDCl$_3$)

$^{19}$F NMR (376 MHz, CDCl$_3$)
$^{13}$C NMR (101 MHz, CDCl$_3$)

$^1$H NMR (400 MHz, CDCl$_3$)
$^{19}$F NMR (376 MHz, CDCl₃)

$^{13}$C NMR (101 MHz, CDCl₃)
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$^{19}\text{F NMR (376 MHz, CDCl}_3\text{)}$

$^{13}\text{C NMR (101 MHz, CDCl}_3\text{)}$
$^1$H NMR (400 MHz, CDCl$_3$)

$^{19}$F NMR (376 MHz, CDCl$_3$)
$^1$$^3$C NMR (101 MHz, CDCl₃)

$^1$$^H$ NMR (400 MHz, CDCl₃)
$^{19}F$ NMR (376 MHz, CDCl$_3$)

$^{13}C$ NMR (101 MHz, CDCl$_3$)
$^1$H NMR ($400 \text{ MHz, CDCl}_3$)

$^{19}$F NMR ($376 \text{ MHz, CDCl}_3$)
$^{13}\text{C NMR (101 MHz, CDCl}_3\text{)}$

$^{1}\text{H NMR (400 MHz, CDCl}_3\text{)}$
$^{19}F$ NMR (376 MHz, $CDCl_3$)

$^{13}C$ NMR (101 MHz, $CDCl_3$)

S35
$^1H$ NMR (400 MHz, $(CD_3)_2CO$)

$^{19}F$ NMR (376 MHz, $(CD_3)_2CO$)
$^{13}$C NMR (101 MHz, (CD$_3$)$_2$CO)

$^1$H NMR (400 MHz, CDCl$_3$)
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$^{19}$F NMR (376 MHz, CDCl$_3$)
$^{13}$C NMR (101 MHz, CDCl$_3$)

$^1$H NMR (400 MHz, CDCl$_3$)
$^{19}\text{F NMR (376 MHz, CDCl}_3\text{)}$

![F NMR spectrum]

$^{13}\text{C NMR (101 MHz, CDCl}_3\text{)}$

![C NMR spectrum]
$^1$H NMR (400 MHz, CDCl₃)

$^{19}$F NMR (376 MHz, CDCl₃)
$^{13}$C NMR (101 MHz, CDCl$_3$)

$^1$H NMR (400 MHz, CDCl$_3$)
$^{19}\text{F NMR (376 MHz, CDCl}_3\text{)}$

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$^{19}$F NMR (376 MHz, CDCl$_3$)
$^{13}$C NMR (101 MHz, CDCl$_3$)

$^1$H NMR (400 MHz, CDCl$_3$)
$^{19}$F NMR (376 MHz, CDCl$_3$)

$^{13}$C NMR (101 MHz, CDCl$_3$)
$^1$H NMR (400 MHz, CDCl$_3$)

$^{19}$F NMR (376 MHz, CDCl$_3$)
$^{13}$C NMR (101 MHz, CDCl$_3$)