Supporting information

A Carbon Isotope Labeling Strategy for β-Amino Acid Derivatives via Carbonylation of Azanickellacycles

Anne K. Ravn†, Maria B. T. Vilstrup†, Peter Noerby‡, Dennis U. Nielsen†*, Kim Daasbjerg†, Troels Skrydstrup†*

†Carbon Dioxide Activation Center (CADIAC), The Interdisciplinary Nanoscience Center (iNANO) and Department of Chemistry, Aarhus University, Gustav Wieds Vej 14, 8000 Aarhus, Denmark

‡Center for Materials Crystallography, Department of Chemistry, Aarhus University, Langelandsgade 140, 8000 Aarhus, Denmark

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General methods
All purchased chemicals, including the organozinc reagents, were used as received without further purification. Solvents were dried according to standard procedures and degassed by bubbling with argon for minimum 30 minutes. Flash column chromatography was carried out on silica gel 60 (230-400 mesh). The $^1$H NMR spectra were recorded at 400 MHz, $^{13}$C NMR spectra were recorded at 100 MHz, and $^{19}$F NMR spectra were recorded at 367 MHz on a Bruker 400 spectrometer. The $^{13}$C NMR spectra of the CO-complexes and of the Sitagliptin derivative were recorded at 126 MHz on a Bruker 500 spectrometer. The chemical shifts are reported in ppm relative to solvent residual peak. Coupling patterns in the NMR spectra are abbreviated as follows: s = singlet, d = doublet, t = triplet, q = quartet, quin = quintuplet, sext = sextet, sep = septet, m = multiplet, br = broad, dd = double doublet, dt = double triplet, ddd = double double doublet, ddt = double double triplet. NMR spectra are reported as follows: (multiplicity; coupling constant(s) in Hz; integration). HRMS spectra were recorded on a LC TOF (ES) apparatus. Melting points were measured on a Büchi B-540 apparatus and optical rotations were measured on a BS ADP440$^+$ polarimeter.

Handling of carbon monoxide
All carbonylation reactions were performed in a two-chamber system, in which gaseous CO was released in one chamber and utilized in a second chamber. The two-chamber system (COware$^\text{®}$)$^1$ is depicted to the right and is composed of two glass vials (Chamber A and B) connected with a glass tube to allow gas-transfer (total volume = 20 mL). The chambers can be sealed with a screw cap and a Teflon$^\text{®}$ coated silicone seal. CO was released from 9-methyl-$9H$-fluorene-$9H$-carbonyl chloride (COgen)$^2$ in a Pd-catalyzed decarbonylation reaction in DMF at 20 °C. Precise conditions are given in the general procedures.
WARNING: Glassware under pressure!
- Glass equipment should always be examined for damages to its surface, which may weaken its strength.
- One must abide to all laboratory safety procedures and always work behind a shield when working with glass equipment under pressure.
- COware is pressure tested to 224 psi, but should under no circumstances be operated above 60 psi (5 bar).

Electrochemical Oxidation of Complex 12

Cyclic voltammetry
The experiment was performed in a sealed flask using a three-electrode setup. The working electrode was a carbon paper electrode, the counter electrode was a Pt mesh and Ag/AgCl was used as reference electrode. The continuous cyclic voltammogram was run between -0.4 V and 0.4 V vs ferrocene/ferrocene* in 0.2 M TBABF$_4$ in dichloroethane. The voltammetric recordings were performed with a CHI 601D potentiostat.

**A:  **

**B:**

**Figure S1:** A) Cyclic voltammogram (V vs ferrocene/ferrocene*) of complex 12 at oxidizing potential. B) Experimental setup using a three-electrode setup in a sealed flask.
Figure S2: Experimental data from the oxidative electrochemical transformation of complex 12 to β-lactam 40. A) Experimental setup. B) Current over time at a constant potential of 0.3 V vs ferrocene/ferrocene*. 
X-ray crystallography data

Crystallographic single crystal X-ray data were collected on a Bruker Kappa Apex2 diffractometer equipped with a Ag micro-focus source. Absorption correction was done with SADABS. Cell refinement and data reduction were done in SAINT-plus. The structures were solved and refined with SHELXT and SHELXL, respectively, in Olex2.

Table with crystallographic information for 1 (left) and 12 (right).
Synthesis of Aziridines

General Procedure 1A\(^6\)
To a solution of chloramine-T trihydrate (1.6 g, 5.5 mmol, 1.1 equiv) and alkene (5.0 mmol, 1.0 equiv) in CH\(_3\)CN (25.0 mL) was added PhNMe\(_3\)Br\(_3\) (188 mg, 0.50 mmol, 10 mol%). The reaction was stirred vigorously at room temperature for 18 h followed by concentration in vacuo. The resulting residue was dissolved in CH\(_2\)Cl\(_2\) (10 mL) and filtered through a short silica column eluting with 10% EtOAc in pentane (200 mL). After evaporation of the solvent, the residue was dissolved in CH\(_3\)CN (10 mL). K\(_2\)CO\(_3\) (2.8 g, 20.0 mmol, 4.0 equiv) was then added and the mixture was stirred for 2 h at 45 °C. After cooling the reaction to room temperature, the mixture was diluted with Et\(_2\)O (20 mL). The mixture was filtered through celite and washed with Et\(_2\)O. The product was purified by flash column chromatography.

General Procedure 1B\(^7\)
A solution of amino alcohol (5.0 mmol, 1.0 equiv) in CH\(_2\)Cl\(_2\) (4 mL) and pyridine (2 mL, 24.8 mmol, 5.0 equiv) was cooled to 0 °C in an ice bath. The corresponding sulfonyl chloride (15.0 mmol, 3.0 equiv) was added in portions and the reaction mixture was stirred for 1 h, before allowed to reach room temperature and stirred for 16 h. The reaction mixture was then diluted with CH\(_2\)Cl\(_2\) (10 mL) and washed with 2 M HCl\(_{(aq)}\). The aqueous phase was extracted with CH\(_2\)Cl\(_2\). The combined organic phases were washed with 2 M KOH\(_{(aq)}\) and the aqueous phase was extracted with additional CH\(_2\)Cl\(_2\). The combined organic phases were dried over Na\(_2\)SO\(_4\), filtered and concentrated in vacuo. The product was purified by flash column chromatography.

General Procedure 1C\(^8\)
Amino alcohol (5.0 mmol, 1.0 equiv) was added to a flame-dried round-bottomed flask and dissolved in dry CH\(_2\)Cl\(_2\) (20 mL) followed by cooling to 0 °C. Et\(_3\)N (2.1 mL, 15 mmol, 3.0 equiv) and DMAP (31 mg, 0.25 mmol, 5.0 mol%) were added to the mixture before TsCl (2.4 g, 12.5 mmol, 2.5 equiv) was added in portions. The reaction mixture was stirred for 18 h at room temperature and quenched with saturated NH\(_4\)Cl\(_{(aq)}\). The aqueous phase was extracted with CH\(_2\)Cl\(_2\) and the combined organic phases were washed with brine and dried over Na\(_2\)SO\(_4\), filtered and concentrated in vacuo. The product was purified by flash column chromatography.
General Procedure 2 (Synthesis of nickellacycles 1-11)\textsuperscript{9}

To a 10 mL glass flask, in an argon filled glovebox, were added Ni(cod)$_2$ (137 mg, 0.50 mmol, 1.0 equiv) and 1,10-phenanthroline (99 mg, 0.55 mmol, 1.1 equiv) which were dissolved in THF (3 mL) and stirred for 5 min before the aziridine (0.60 mmol, 1.2 equiv) was added. The reaction mixture was sealed using a screw cap equipped with a Teflon coated seal before being removed from the glovebox. The reaction was stirred for 30 min at room temperature before being stirred at 60 °C for 3 h. The reaction was cooled to room temperature before being transferred to an argon-filled glovebox where pentane or diethyl ether (4 mL) was added followed by vigorous stirring for 1 h at room temperature. The reaction was filtered and the solid was washed with pentane or diethyl ether before being dried \textit{in vacuo}. Melting points could not be obtained as the complexes decomposed around 200 °C.

General Procedure 3 (Synthesis of acyl nickeallacycles 12-22)

\textit{Chamber 1:} In an argon filled glovebox, to chamber 1 of the 2-chamber reactor (100 mL), was added azanickellacyclobutane complex (0.50 mmol, 1 equiv) and dichloroethane (5 mL). The chamber was sealed with a screwcap fitted with a Teflon® coated seal.

\textit{Chamber 2 (1.5 equiv $^{13}$CO):} In an argon filled glovebox, to chamber 2 of the 2-chamber reactor, was added $^{13}$C-COgen (183.3 mg, 0.75 mmol, 1.5 equiv), Pd(dba)$_2$ (21.9 mg, 0.038 mmol, 5 mol%), HBF$_4P(tBu)$_3 (21.8 mg, 0.075 mmol, 10 mol%), DMF (5 mL) and Cy$_2$NMe ($321 \mu$L, 1.5 mmol, 2 equiv) in that order. The chamber was sealed with a screwcap fitted with a Teflon® coated seal. The reaction was stirred at room temperature for 24 h. After the designated time, the complex had either 1) precipitated in solution and could be isolated by filtration after being stirred in pentane in the glovebox or 2) the solution was concentrated \textit{in vacuo} in an argon filled glovebox and then precipitated from dichloroethane using pentane. The desired product was isolated by filtration, washed with pentane and dried \textit{in vacuo}.

The complexes are unstable in solution towards air over prolonged time and the $^{13}$C-enriched signals in the range 160-220 ppm in $^{13}$C NMR are products of decomposition or reductive elimination. The $^{13}$C NMR were recorded at 126 MHz on a Bruker 500 spectrometer, however, not all signals were observed for all the complexes. The complexes are stable in air as solids. Complex 12 was stored under atmospheric air for 14 days. The NMR analysis of before and after
are comparable. Both NMR analyses are attached. Melting points could not be obtained as the complexes decomposed around 200 °C.

**General Procedure 4 (Synthesis of methyl esters 23-33)**

In an argon filled glovebox complex (12-22) (0.2 mmol, 1 equiv) was dissolved in dichloroethane (5 mL) in a 10 mL flask. The loaded glassware was removed from the glovebox and 4 M HCl\(_{\text{aq}}\) (2 mL) was added under atmospheric air followed by stirring at room temperature for 1 h. Formic acid (2 mL) was added and the reaction was stirred for additional 4 h. The reaction was diluted with dichloromethane and the aqueous phase was extracted with dichloromethane. The combined organic phases were washed with brine, dried over Na\(_2\)SO\(_4\), concentrated *in vacuo*. The crude was dissolved in a mixture of Et\(_2\)O (5 mL) and MeOH (1 mL) and cooled to 0 °C. TMSCHN\(_2\) (0.2 mL, 0.40 mmol, 2.0 equiv, 2.0 M in Et\(_2\)O) was slowly added and the mixture was stirred for 1 h followed by quenching with aqueous acetic acid (33%). The aqueous phase was extracted with Et\(_2\)O (3x) and the combined organic phases were dried over MgSO\(_4\), concentrated *in vacuo* and purified by flash column chromatography.

**General Procedure 5 (Synthesis of ketones 34-36)**

12 (28 mg, 0.05 mmol, 1.0 equiv) was dissolved in DMA (1 mL) in an argon filled glovebox in a 10 mL flask. The corresponding alkylzinc bromide (0.15 mmol, 3.0 equiv) was added dropwise under stirring and the reaction sealed with a screw cap fitted with a Teflon ® seal and removed from the glovebox and stirred at room temperature for 16 h. The reaction was diluted with Et\(_2\)O and quenched with 2M HCl\(_{\text{aq}}\). The mixture was extracted with Et\(_2\)O and the combined organic phases were dried over Na\(_2\)SO\(_4\), filtered and concentrated *in vacuo*. The products were isolated by flash column chromatography.
Characterization of products

2-Benzyl-1-tosylaziridine (Compound 1a)

1a was prepared according to General Procedure 1A employing allylbenzene (591 mg, 5.0 mmol). Flash column chromatography (10% EtOAc in pentane) afforded the product as a colorless solid (759 mg, 53%). The spectral data was in accordance with literature.\(^\text{10}\) mp: 62–66 °C, mp(lit): 67–70 °C.\(^\text{11}\) \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.68 (d, \(J = 8.2 \) Hz, 2 H), 7.21 (d, \(J = 8.1 \) Hz, 2 H), 7.18–7.12 (m, 3 H), 7.04 (dd, \(J = 6.3, 2.7 \) Hz, 2 H), 2.99–2.91 (m, 1 H), 2.81 (dd, \(J = 14.5, 5.2 \) Hz, 1 H), 2.73–2.65 (m, 2 H), 2.42 (s, 3 H), 2.16 (d, \(J = 4.5 \) Hz, 1 H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) (ppm): 144.4, 137.1, 134.9, 129.7 (2 C), 128.8 (2 C), 128.6 (2 C), 128.0 (2 C), 126.6, 41.3, 37.6, 33.0, 21.8. HRMS C\(_{16}\)H\(_{17}\)NO\(_2\)S [M+H\(^+\)]; calculated 288.1053, found 288.1054.

(S)-2-Benzyl-1-tosylaziridine (Compound 1a’)

1a’ was prepared according to General Procedure 1C employing (S)-phenylalaninol (756 mg, 5.0 mmol). Flash column chromatography (10% EtOAc in pentane) afforded the product as a colorless solid (753 mg, 53%). The physical and spectral data were in accordance with literature.\(^\text{12}\) [\(\alpha\)]\(_{23}\)\(^D\) = 5.6 (c = 0.5, CHCl\(_3\)), Lit: [\(\alpha\)]\(_{25}\)\(^D\) = 14.8 (c = 0.4, CHCl\(_3\)). mp: 92–93 °C, (lit, 86–88 °C). The NMR and HRMS data were identical with that of 1a.

2-(Benza[d][1,3]dioxol-5-ylmethyl)-1-tosylaziridine (Compound 2a)

The title compound was prepared according to General Procedure 1A employing safrole (811 mg, 5.0 mmol). Flash column chromatography (15% EtOAc in pentane) afforded the product as a colorless oil (773 mg, 47%). The spectral data was in accordance with literature.\(^\text{13}\) \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.68 (d, \(J = 8.2 \) Hz, 2 H), 7.23 (d, \(J = 8.0 \) Hz, 2 H), 6.58 (d, \(J = 6.8 \) Hz, 1 H), 6.51–6.46 (m, 2 H), 5.92–5.88 (m, 2 H), 2.87 (tt, \(J = 7.1, 4.6 \) Hz, 1 H), 2.76 (dd, \(J = 14.5, 4.8 \) Hz, 1 H), 2.72 (d, \(J = 6.9 \) Hz, 1 H), 2.52 (dd, \(J = 14.5, 7.5 \) Hz, 1 H), 2.43 (s, 3 H), 2.15 (d, \(J = 4.5 \) Hz, 1 H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) (ppm): 147.6, 146.4, 144.5, 135.0, 130.9, 129.6 (2 C), 128.0 (2 C), 121.8, 109.3, 108.3, 101.0, 41.6, 37.3, 32.8, 21.7. HRMS C\(_{17}\)H\(_{17}\)NO\(_4\)S [M+H\(^+\)]; calculated 332.0951, found 332.0952.
2-Phenyl-1-tosylaziridine (Compound 3a)

The title compound was prepared according to General Procedure 1A employing styrene (521 mg, 5.0 mmol). Flash column chromatography (10% EtOAc in pentane) afforded the product as a colorless solid (752 mg, 55%). The spectral data was in accordance with literature.\(^\text{13}\) mp: 89–90 °C, mp(litt): 90–91 °C.\(^\text{14}\) \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.87 (d, \(J = 8.2\) Hz, 2 H), 7.33 (d, \(J = 8.2\) Hz, 2 H), 7.30–7.27 (m, 3 H), 7.22 (dd, \(J = 7.1, 2.1\) Hz, 2 H), 3.78 (dd, \(J = 7.1, 4.4\) Hz, 1 H), 2.99 (d, \(J = 7.2\) Hz, 1 H), 2.44 (s, 3 H), 2.39 (d, \(J = 4.5\) Hz, 1 H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) (ppm): 144.8, 135.2, 135.2, 129.9 (2 C), 128.7 (2 C), 128.4, 128.1 (2 C), 126.7 (2 C), 41.2, 36.1, 21.8. HRMS C\(_{15}\)H\(_{15}\)NO\(_2\)S [M+H\(^+\)]; calculated 274.0896, found 274.0898.

2-Methyl-1-tosylaziridine (Compound 4a)

\[
\text{Ts} \quad \begin{array}{c} \text{N} \\ \text{I} \end{array} 
\]

The title compound was prepared according to General Procedure 1C employing alaninol (376 mg, 5.0 mmol). Flash column chromatography (5 → 20% EtOAc in pentane) afforded the product as a colorless solid (847 mg, 80%). The spectral data was in accordance with literature.\(^\text{9}\) mp: 58–62 °C, mp(litt): 58 °C.\(^\text{15}\) \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.83 (d, \(J = 8.1\) Hz, 2 H), 7.34 (d, \(J = 8.0\) Hz, 2 H), 2.83 (sext, \(J = 6.6\) Hz, 1 H), 2.62 (d, \(J = 7.0\) Hz, 1 H), 2.45 (s, 3 H), 2.02 (d, \(J = 4.6\) Hz, 1 H), 1.26 (d, \(J = 5.6\) Hz, 3 H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) (ppm): 144.8, 135.2, 135.2, 129.9 (2 C), 128.7 (2 C), 128.4, 128.1 (2 C), 126.7 (2 C), 36.0, 34.9, 21.8, 16.9. HRMS C\(_{10}\)H\(_{13}\)NO\(_2\)S [M+H\(^+\)]; calculated 212.0740, found 212.0740.

2-Isopropyl-1-tosylaziridine (Compound 5a)

\[
\text{Ts} \quad \begin{array}{c} \text{N} \\ \text{I} \end{array} 
\]

The title compound was prepared according to General Procedure 1C employing valinol (516 mg, 5.0 mmol). Flash column chromatography (5 → 8% EtOAc in pentane) afforded the product as a colorless solid (827 mg, 69%). The physical and spectral data was in accordance with literature.\(^\text{16}\) mp: 62–64 °C, mp(litt): 66–68 °C.\(^\text{1}\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.83 (d, \(J = 8.2\) Hz, 2 H), 7.34 (d, \(J = 8.0\) Hz, 2 H), 2.62 (d, \(J = 7.0\) Hz, 1 H), 2.52 (td, \(J = 7.3, 4.6\) Hz, 1 H), 2.45 (s, 3 H), 2.10 (d, \(J = 4.6\) Hz, 1 H), 1.41 (dq, \(J = 13.8, 6.9\) Hz, 1 H), 0.90 (d, \(J = 6.8\) Hz, 3 H), 0.80 (d, \(J = 6.7\) Hz, 3 H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) (ppm): 144.5, 135.3, 129.7 (2 C), 128.2 (2 C), 46.4, 32.9, 30.3, 21.8, 19.7, 19.2. HRMS C\(_{12}\)H\(_{17}\)NO\(_2\)S [M+Na\(^+\)]; calculated 262.0872, found 262.0863.
2-(Cyclohexylmethyl)-1-tosylaziridine (Compound 6a)

The title compound was prepared according to General Procedure 1A employing allylcyclohexane (621 mg, 5.0 mmol). Flash column chromatography (20 → 40% EtOAc in pentane) afforded the product as a colorless oil (842 mg, 57%). $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 7.83 (d, $J$ = 8.2 Hz, 2 H), 7.33 (d, $J$ = 8.0 Hz, 2 H), 2.76 (app $\text{tt}$, $J$ = 7.2, 5.1 Hz, 1 H), 2.65 (d, $J$ = 7.0 Hz, 1 H), 2.45 (s, 3 H), 2.02 (d, $J$ = 4.6 Hz, 1 H), 1.69–1.53 (m, 5 H), 1.38–1.25 (m, 2 H), 1.23–1.15 (m, 1 H), 1.13–1.01 (m, 3 H), 0.90–0.79 (m, 2 H).

$^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm): 144.5, 135.3, 129.7 (2 C), 128.1 (2 C), 80.1, 79.8, 67.9, 67.6, 48.3, 48.2, 40.5, 40.2, 39.4 (2 C), 34.6 (2 C), 31.6 (2 C), 31.3 (2 C), 25.8, 25.5, 23.5, 23.4, 22.4 (2 C), 21.8 (2 C), 21.1, 21.0, 16.4, 16.3. HRMS C$_{16}$H$_{23}$NO$_2$S [M+H$^+$]; calculated 294.1522, found 294.1529.

2-(((1R,2S,5R)-2-Isoproyl-5-methylcyclohexyl)oxy)methyl)-1-tosylaziridine (Compound 7a)

The title compound was prepared according to General Procedure 1A employing (1S,2R,4R)-2-(allyloxy)-1-isopropyl-4-methylcyclohexane (982 mg, 5.0 mmol). Flash column chromatography (5% EtOAc in pentane) afforded the product as a colorless oil (621 mg, 34%). The compound was isolated as a mixture of diastereomers (1:1). $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 7.83 (d, $J$ = 8.2 Hz, 4 H), 7.33 (d, $J$ = 7.9 Hz, 4 H), 3.60 (dt, $J$ = 10.1, 4.7 Hz, 2 H), 3.34 (dt, $J$ = 10.6, 4.5 Hz, 2 H), 3.02–2.92 (m, 4 H), 2.67 (dd, $J$ = 7.0, 3.6 Hz, 2 H), 2.45 (s, 6 H), 2.20 (dd, $J$ = 13.8, 4.5 Hz, 2 H), 2.09–1.88 (m, 4 H), 1.65–1.55 (m, 4 H), 1.34–1.20 (m, 4 H), 1.16–1.04 (m, 2 H), 0.89 (d, $J$ = 4.8 Hz, 6 H), 0.86 (d, $J$ = 3.6 Hz, 4 H), 0.84 (bs, 2 H), 0.81 (d, $J$ = 7.2 Hz, 4 H), 0.71 (d, $J$ = 7.2 Hz, 3 H), 0.66 (d, $J$ = 6.8 Hz, 3 H).

$^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm): 144.5 (2 C), 135.3, 135.2, 129.7 (4 C), 128.2 (2 C), 128.1 (2 C), 80.1, 79.8, 67.9, 67.6, 48.3, 48.2, 40.5, 40.2, 39.4 (2 C), 34.6 (2 C), 31.6 (2 C), 31.3 (2 C), 25.8, 25.5, 23.5, 23.4, 22.4 (2 C), 21.8 (2 C), 21.1, 21.0, 16.4, 16.3. HRMS C$_{20}$H$_{31}$NO$_3$S [M+H$^+$]; calculated 366.2097, found 366.2100.
2-Benzyl-1-((4-methoxyphenyl)sulfonyl)aziridine (Compound 8a)

The title compound was prepared according to General Procedure 1B employing allylbenzene (591 mg, 5.0 mmol) and 4-methoxybenzenesulfonyl chloride (1.0 g, 15.0 mmol). Flash column chromatography (5 → 15% EtOAc in pentane) afforded the product as a colorless solid (1.2 g, 79%). The spectral data was in accordance with literature.\(^{17}\) mp: 71–74 °C. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.72 (d, \(J = 9.0\) Hz, 2 H), 7.18–7.13 (m, 3 H), 7.07–7.02 (m, 2 H), 6.87 (d, \(J = 8.9\) Hz, 2 H), 3.87 (s, 3 H), 2.96–2.89 (m, 1 H), 2.82 (dd, \(J = 14.4\), 5.0 Hz, 1 H), 2.72–2.63 (m, 2 H), 2.16 (d, \(J = 4.4\) Hz, 1 H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) (ppm): 163.6, 137.2, 130.1 (2 C), 129.5, 128.8 (2 C), 128.6 (2 C), 126.7, 114.3 (2 C), 55.7, 41.3, 37.7, 32.9. HRMS C\(_{16}\)H\(_{17}\)NO\(_2\)S [M+H\(^{+}\)]; calculated 304.1002, found 304.1010.

2-Benzyl-1-((methylsulfonyl)aziridine (Compound 9a)

The title compound was prepared according to General Procedure 1B employing allylbenzene (591 mg, 5.0 mmol) and methanesulfonyl chloride (1.7 g, 15.0 mmol). The product was isolated as an off-white solid (1.0 g, 98%) after work-up. Flash column chromatography was not conducted. The spectral data was in accordance with literature.\(^{18}\) mp: 54–56 °C. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.36–7.31 (m, 2 H), 7.29–7.24 (m, 3 H), 2.99 (dd, \(J = 14.0\), 4.8 Hz, 1 H), 2.91 (app tt, \(J = 7.5\), 4.6 Hz, 1 H), 2.73 (s, 3 H), 2.69 (dd, \(J = 13.1\), 7.2 Hz, 2 H), 2.22 (d, \(J = 4.5\) Hz, 1 H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) (ppm): 137.3, 129.0 (2 C), 128.8 (2 C), 127.1, 41.3, 39.3, 37.6, 32.6. HRMS C\(_{10}\)H\(_{13}\)NO\(_2\)S [M+Na\(^{+}\)]; calculated 234.0559, found: 234.0562.

2-Benzyl-1-((4-trifluoromethylphenyl)sulfonyl)aziridine (Compound 10a)

The title compound was prepared according to General Procedure 1B employing allylbenzene (591 mg, 5.0 mmol) and 4-(trifluoromethyl)benzenesulfonyl chloride (1.2 g, 15.0 mmol). The product was isolated as a colorless solid (1.3 g, 77%). mp: 102–103 °C. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.85 (d, \(J = 8.2\) Hz, 2 H), 7.62 (d, \(J = 8.3\) Hz, 2 H), 7.16-7.06 (m, 3 H), 6.97 (d, \(J = 7.1\) Hz, 2 H), 3.05–2.97 (m, 1 H), 2.94 (dd, \(J = 14.3\), 4.3 Hz, 1 H), 2.85 (d, \(J = 6.9\) Hz, 1 H), 2.51 (dd, \(J = 14.2\), 8.1 Hz, 1 H), 2.27 (d, \(J = 4.5\) Hz, 1 H). \(^{13}\)C NMR (100 MHz,
CDCl$_3$ $\delta$ (ppm): 141.5 (q, $J$ = 1.1 Hz), 137.0, 135.0 (q, $J$ = 33.0 Hz), 128.7 (2 C), 128.6 (2 C), 128.4 (2 C), 127.0, 126.1 (q, $J$ = 3.7 Hz, 2 C), 123.3 (q, $J$ = 271.4 Hz), 42.7, 37.7, 33.2. $^{19}$F NMR (367 MHz, CDCl$_3$) $\delta$ (ppm): -63.3. HRMS C$_{16}$H$_{14}$F$_3$NO$_2$S $\left[\text{M+H}^+\right]$; calculated 342.0770, found 342.0771.

2-Benzyl-1-((trifluoromethyl)sulfonyl)aziridine (Compound 11a)

2-Amino-3-phenylpropan-1-ol (1.5 g, 10.0 mmol, 1.0 equiv) was dissolved in dry CH$_2$Cl$_2$ (40 mL) in a flame-dried round-bottomed flask before Et$_3$N (2.8 mL, 20.0 mmol, 2.0 equiv) was added. The mixture was then cooled to -78 °C. Trifluoromethanesulfonic anhydride (3.7 mL, 22.0 mmol, 2.2 equiv) was added dropwise over 1 h. The reaction mixture was stirred for 18 h slowly allowing to reach room temperature. The mixture was then diluted with CH$_2$Cl$_2$ (40 mL) and washed with first 0.1 M HCl(aq) and then saturated NaHCO$_3$(aq). The combined organic phases were dried over MgSO$_4$, filtered, and concentrated in vacuo. The residue was filtered over celite eluting with CH$_2$Cl$_2$ (100 mL) to afford the title compound (2.6 g, 96%) as a colorless oil.

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ (ppm): 7.38 – 7.28 (m, 3 H), 7.21 (d, $J$ = 7.4 Hz, 2 H), 3.31 (quin, $J$ = 5.8 Hz, 1 H), 3.05 (dd, $J$ = 14.6, 5.7 Hz, 1 H), 2.93 (d, $J$ = 6.5 Hz, 1 H), 2.89 (d, $J$ = 6.5 Hz, 1 H), 2.45 (d, $J$ = 4.9 Hz, 1 H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ (ppm): 135.2, 129.1 (2 C), 128.9 (2 C), 127.5, 119.1 (q, $J$ = 320.7 Hz), 42.6, 37.1, 35.0. $^{19}$F NMR (367 MHz, CDCl$_3$) $\delta$ (ppm): -76.6. HRMS C$_{10}$H$_{10}$F$_3$NO$_2$S$^+$ [M+H$^+$]; calculated 266.0457, found 266.0451.

(Phen)Ni{N(Ts)CH(Bn)CH$_2$} and (S)-(Phen)Ni{N(Ts)CH(Bn)CH$_2$} (Compounds 1 and 1’)

The title compounds were prepared according to General Procedure 2 employing 2-benzyl-1-tosylaziridine (1a) or (S)-2-benzyl-1-tosylaziridine (1a’) (172 mg, 0.60 mmol) yielding the desired products as a dark purple solids (1, 203 mg, 77%), (1’, 208 mg, 79%). $^1$H NMR (400 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 9.97 (d, $J$ = 4.8 Hz, 1 H), 8.46 (d, $J$ = 8.1 Hz, 1 H), 8.40 (d, $J$ = 8.1 Hz, 1 H), 8.31–8.24 (m, 3 H), 7.96–7.84 (m, 3 H), 7.53 (dd, $J$ = 8.1, 5.2 Hz, 1 H), 7.25–7.17 (m, 6 H), 7.16–7.09 (m, 1 H), 3.95 (ddt, $J$ = 12.0, 8.3, 4.1 Hz, 1 H), 3.02 (dd, $J$ = 13.0, 3.8 Hz, 1 H), 2.81 (dd, $J$ = 13.0, 10.4 Hz, 1 H), 2.35 (s, 3 H), 0.48 (dd, $J$ = 8.3, 5.5 Hz, 1 H), 0.35 (app t, $J$ = 5.0 Hz, 1 H). $^{13}$C NMR (100 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 153.2, 149.3, 147.1, 144.3, 143.2, 140.8, 140.3,
The title compound was prepared according to General Procedure 2 employing 2-(benzo[d][1,3]dioxol-5-ylmethyl)-1-tosylaziridine (2a) (199 mg, 0.6 mmol) yielding the desired product as a dark red solid (205 mg, 72%) with minor THF impurities. 

$^1$H NMR (400 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 9.95 ($d$, $J = 4.9$ Hz, 1 H), 8.46 ($d$, $J = 8.1$ Hz, 1 H), 8.40 ($d$, $J = 8.0$ Hz, 1 H), 8.28 ($d$, $J = 5.4$ Hz, 1 H), 8.25 ($d$, $J = 8.1$ Hz, 2 H), 7.95–7.84 (m, 3 H), 7.54 ($dd$, $J = 8.1$, 5.3 Hz, 1 H), 7.21 ($d$, $J = 7.9$ Hz, 2 H), 6.72 (s, 1 H), 6.66 (s, 2 H), 5.84 ($d$, $J = 5.5$ Hz, 2 H), 3.92–3.84 (m, 1 H), 2.73 ($dd$, $J = 12.9$, 10.3 Hz, 1 H), 2.35 (s, 3 H), 0.48 ($dd$, $J = 8.1$, 5.4 Hz, 1 H), 0.32 (app t, $J = 4.9$ Hz, 1 H). 

$^{13}$C NMR (100 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 153.2, 149.4, 147.7, 147.2, 146.0, 144.3, 143.3, 140.9, 137.0, 136.2, 134.3, 130.0 (2 C), 129.2, 128.7, 127.7 (2 C), 127.4, 126.4 (2 C), 125.4, 122.7, 110.4, 108.1, 101.2, 67.1, 46.2, 21.5, -6.1.

The title compound was prepared according to General Procedure 2 employing 2-phenyl-1-tosylaziridine (3a) (137 mg, 0.6 mmol) yielding the desired product as a dark purple solid (225 mg, 88%). 

$^1$H NMR (400 MHz, CD$_2$Cl$_2$) $\delta$ (ppm) 9.95 ($d$, $J = 4.5$ Hz, 1 H), 8.47 ($d$, $J = 4.9$ Hz, 1 H), 8.42 ($d$, $J = 8.2$ Hz, 1 H), 8.30 ($d$, $J = 8.0$ Hz, 1 H), 8.13 ($d$, $J = 8.1$ Hz, 2 H), 7.90–7.83 (m, 4 H), 7.78 ($d$, $J = 8.9$ Hz, 1 H), 7.49 ($dd$, $J = 8.1$, 5.4 Hz, 1 H), 7.26 ($d$, $J = 7.9$ Hz, 2 H), 7.10–7.04 (m, 3 H), 4.13 ($dd$, $J = 10.7$, 8.6 Hz, 1 H), 3.78 ($dd$, $J = 10.8$, 5.2 Hz, 1 H), 2.40 (s, 3 H), 1.86 ($dd$, $J = 8.7$, 5.2 Hz, 1 H). 

$^{13}$C NMR (100 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 153.7, 151.4, 150.3, 146.8, 143.8, 142.2, 141.1, 137.1, 136.2, 129.4 (2 C), 129.1 (2 C), 128.7, 127.7 (2 C), 127.6 (2 C), 127.4, 126.4, 126.1, 125.5, 123.8, 62.0, 21.6, 4.8. One carbon signal is missing.
(Phen)Ni{N(Ts)CH(Me)CH₂} (Compound 4)

The title compound was prepared according to General Procedure 2 employing 2-methyl-1-tosylaziridine (4a) (127 mg, 0.60 mmol) yielding the desired product as a dark purple solid (199 mg, 89%). ¹H NMR (400 MHz, CD₂Cl₂) δ (ppm): 9.95 (d, J = 4.8 Hz, 1 H), 8.45 (d, J = 7.8 Hz, 1 H), 8.40 (d, J = 8.3 Hz, 1 H), 8.32 (d, J = 5.2 Hz, 1 H), 8.26 (d, J = 8.1 Hz, 2 H), 7.95–7.84 (m, 3 H), 7.57 (dd, J = 7.9, 5.3 Hz, 1 H), 7.21 (d, J = 8.1 Hz, 2 H), 3.92–3.83 (m, 1 H), 2.36 (s, 3 H), 1.20 (d, J = 6.3 Hz, 3 H), 0.75 (dd, J = 8.3, 5.1 Hz, 1 H), 0.21 (app t, J = 4.8 Hz, 1 H). ¹³C NMR (100 MHz, CD₂Cl₂) δ (ppm): 153.3, 149.3, 147.2, 144.3, 143.6, 140.7, 137.0, 136.1, 130.0, 129.3, 129.2 (2 C), 128.7, 127.6 (2 C), 127.5, 126.4, 125.4, 61.6, 26.8, 21.5, -3.1.

(Phen)Ni{N(Ts)CH(isopropyl)CH₂} (Compound 5)

The title compound was prepared according to General Procedure 2 employing 2-isopropyl-1-tosylaziridine (5a) (144 mg, 0.60 mmol) yielding the desired product as a dark purple solid (201 mg, 84%) with minor THF impurities. ¹H NMR (400 MHz, CD₂Cl₂) δ (ppm): 9.86 (d, J = 4.9 Hz, 1 H), 8.45 (d, J = 8.2 Hz, 1 H), 8.40 (d, J = 8.1 Hz, 1 H), 8.34 (d, J = 5.2 Hz, 1 H), 8.30 (d, J = 8.1 Hz, 2 H), 7.96–7.82 (m, 3 H), 7.56 (dd, J = 8.1, 5.2 Hz, 1 H), 7.19 (d, J = 8.0 Hz, 2 H), 3.58 (ddd, J = 8.3, 6.3, 4.2 Hz, 1 H), 2.35 (s, 3 H), 1.89–1.76 (m, 1 H), 1.10 (d, J = 6.7 Hz, 3 H), 0.88 (d, J = 6.8 Hz, 3 H), 0.35 (dd, J = 8.6, 5.3 Hz, 1 H), 0.26 (app t, J = 4.7 Hz, 1 H). ¹³C NMR (100 MHz, CD₂Cl₂) δ (ppm): 152.7, 149.1, 147.0, 144.1, 144.0, 140.5, 136.8, 136.1, 129.3, 129.2 (2 C), 128.5, 127.8 (2 C), 127.3, 126.4, 126.2, 125.4, 70.9, 34.9, 21.5, 19.2, 17.5, -9.4.

(Phen)Ni{N(Ts)CH(methylene cyclohexyl)CH₂} (Compound 6)

The title compound was prepared according to General Procedure 2 employing 2-(cyclohexylmethyl)-1-tosylaziridine (6a) (176 mg, 0.60 mmol) yielding the desired product as a dark purple solid (266 mg, 82%). ¹H NMR (400 MHz, CD₂Cl₂) δ (ppm): 9.90 (d, J = 5.0 Hz, 1 H), 8.45 (d, J = 8.1 Hz, 1 H), 8.40 (d, J = 8.1 Hz, 1 H), 8.33 (d, J = 5.2 Hz, 1 H), 8.23 (d, J = 7.9 Hz, 2 H), 7.94–7.83 (m, 3 H), 7.57 (dd, J = 8.1, 5.2 Hz, 1 H), 7.20 (d, J = 7.8 Hz, 2 H), 3.82 (tt, J = 9.2, 4.7 Hz, 1 H), 2.36 (s, 3 H), 1.69–1.57 (m, 5 H), 1.56–1.48 (m, 2 H), 1.35–1.23 (m, 1 H), 1.22–
1.09 (m, 3 H), 0.96–0.76 (m, 2 H), 0.63 (dd, \( J = 7.9, 5.2 \) Hz, 1 H), 0.24 (app t, \( J = 4.7 \) Hz, 1 H).

\(^{13}\)C NMR (100 MHz, CD\(_2\)Cl\(_2\)) \( \delta \) (ppm): 153.1, 149.2, 147.2, 144.3, 143.7, 140.7, 136.9, 136.1, 129.3, 129.1 (2 C), 128.6, 127.8 (2 C), 127.4, 126.4 (2 C), 125.4, 63.8, 48.6, 34.9, 34.5, 33.7, 27.3, 27.0, 26.9, 21.5, -4.6.

\((\text{Phen})\text{Ni}\{\text{N(Ts)CH(3-((1R, 2S, 5R)-2-isopropyl-5-methylcyclohexyl)methoxy})\text{CH}_2\}\)  (Compound 7)

The title compound was prepared according to General Procedure 2 employing \( 2-((\text{(1R, 2S, 5R)-2-isopropyl-5-methylcyclohexyl)oxy})\text{methyl}-1\)-tosylaziridine (7a) (219 mg, 0.60 mmol) yielding the desired product as a dark red solid, isolated as a mixture of diastereomers (1:1) (281 mg, 93%). \(^1\)H NMR (400 MHz, CD\(_2\)Cl\(_2\)) \( \delta \) (ppm): 9.90 (d, \( J = 5.1 \) Hz, 2 H), 8.45 (d, \( J = 8.0 \) Hz, 2 H), 8.40 (d, \( J = 8.1 \) Hz, 2 H), 8.35 (dd, \( J = 8.1, 5.2 \) Hz, 2 H), 8.22 (dd, \( J = 10.7, 8.2 \) Hz, 4 H), 7.95–7.84 (m, 6 H), 7.57 (ddd, \( J = 7.8, 5.2, 1.5 \) Hz, 2 H), 7.21 (d, \( J = 7.8 \) Hz, 4 H), 3.81 (dtd, \( J = 21.5, 8.7, 4.2 \) Hz, 2 H), 3.65–3.64 (m, 1 H), 3.62 (t, \( J = 9.3 \) Hz, 1 H), 3.40–3.33 (m, 2 H), 3.00 (dtd, \( J = 18.1, 10.5, 4.2 \) Hz, 2 H), 2.36 (s, 6 H), 2.32–2.19 (m, 2 H), 2.16–2.05 (m, 2 H), 1.65–1.53 (m, 4 H), 1.38–1.27 (m, 2 H), 1.19–1.07 (m, 2 H), 0.95 (dd, \( J = 12.6, 3.1 \) Hz, 2 H), 0.92–0.85 (m, 7 H), 0.85–0.80 (m, 8 H), 0.77–0.71 (m, 7 H), 0.61–0.56 (m, 2 H), 0.45–0.39 (m, 2 H). \(^{13}\)C NMR (100 MHz, CD\(_2\)Cl\(_2\)) \( \delta \) (ppm): 154.5 (2 C), 150.9, 150.8, 148.6 (2 C), 145.6 (2 C), 144.6, 144.5, 142.3 (2 C), 138.3 (2 C), 137.5 (2 C), 130.7 (2 C), 130.6 (4 C), 130.1 (2 C), 129.1 (4 C), 128.8 (2 C), 127.8 (2 C), 126.8, 126.7, 81.5, 80.7, 76.9, 76.1, 66.8, 66.1, 50.4, 50.4, 42.8, 42.6, 36.6 (2 C), 33.5, 33.4, 27.5, 27.4, 25.3, 25.3, 24.0 (2 C), 22.3 (2 C), 22.7, 22.6, 18.1, 18.0, -5.2, -5.3. Two aromatic carbon signals are missing.
(Phen)Ni{N(4-methoxybenzenesulfonyl)CH(Bn)CH₂} (Compound 8)

The title compound was prepared according to General Procedure 2 employing 2-benzyl-1-((4-methoxyphenyl)sulfonyl)aziridine (8a) (182 mg, 0.60 mmol) yielding the desired product as a dark red solid (202 mg, 75%) with minor impurities of diethyl ether. $^1$H NMR (400 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 9.96 (d, $J = 4.8$ Hz, 1 H), 8.47 (d, $J = 8.2$ Hz, 1 H), 8.40 (d, $J = 8.0$ Hz, 1 H), 8.31 (d, $J = 8.7$, 2 H), 8.28 (d, $J = 5.2$ Hz, 1 H), 7.97–7.84 (m, 3 H), 7.54 (dd, $J = 8.1$, 5.2 Hz, 1 H), 7.28–7.17 (m, 4 H), 7.17–7.09 (m, 1 H), 6.89 (d, $J = 8.7$ Hz, 2 H), 3.98–3.88 (m, 1 H), 3.80 (s, 3 H), 3.01 (dd, $J = 12.9$, 3.7 Hz, 1 H), 2.85–2.77 (m, 1 H), 0.48 (dd, $J = 8.1$, 5.6 Hz, 1 H), 0.35 (app t, $J = 4.9$ Hz, 1 H). $^{13}$C NMR (100 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 161.6, 153.2, 149.4, 147.2, 144.3, 140.5, 138.4, 137.0, 136.2, 130.1 (2 C), 129.6 (2 C), 129.4, 128.7, 128.5 (2 C), 127.4, 126.5, 126.4, 126.1, 125.4, 113.8 (2 C), 67.1, 55.9, 46.6, -6.0.

(Phen)Ni{N(methylsulfonyl)CH(Bn)CH₂} (Compound 9)

The title compound was prepared according to General Procedure 2 employing 2-benzyl-1-(methylsulfonyl)aziridine (9a) (127 mg, 0.60 mmol) yielding the desired product as a dark purple solid (198 mg, 88%) with minor impurities of dichloroethane. $^1$H NMR (400 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 9.76 (d, $J = 4.9$ Hz, 1 H), 8.49–8.36 (m, 3 H), 7.94–7.82 (m, 3 H), 7.58 (dd, $J = 8.1$, 5.3 Hz, 1 H), 7.33–7.24 (m, 4 H), 7.19–7.14 (m, 1 H), 4.15 (tt, $J = 9.1$, 4.6 Hz, 1 H), 3.10 (dd, $J = 13.0$, 4.6 Hz, 1 H), 3.03 (s, 3 H), 2.85 (dd, $J = 13.0$, 9.9 Hz, 1 H), 0.85 (dd, $J = 8.3$, 5.5 Hz, 1 H), 0.49 (app t, $J = 5.1$ Hz, 1 H). $^{13}$C NMR (100 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 153.1, 149.5, 147.2, 144.4, 140.6, 137.0, 136.2, 130.2 (2 C), 129.4, 128.7, 128.5 (2 C), 127.4, 126.5, 126.4, 126.2, 125.4, 67.9, 47.4, 41.6, -5.6.
(Phen)Ni{N(4-trifluoromethylbenzenesulfonyl)CH(Bn)CH₂} (Compound 10)

The title compound was prepared according to General Procedure 2 (the compound was precipitated from THF using toluene instead of pentane) employing 2-benzyl-1-((4-trifluoromethylphenyl)sulfonyl)aziridine (10a) (205 mg, 0.6 mmol) yielding the title compound as a dark purple solid (256 mg, 88%). ¹H NMR (400 MHz, CD₂Cl₂) δ (ppm): 9.89 (d, J = 4.9 Hz, 1 H), 8.53 (d, J = 8.1 Hz, 1 H), 8.47 (d, J = 8.2 Hz, 1 H), 8.40 (d, J = 8.0 Hz, 1 H), 8.23 (d, J = 5.0 Hz, 1 H), 7.98–7.84 (m, 3 H), 7.66 (d, J = 8.1 Hz, 2 H), 7.57–7.51 (m, 1 H), 7.28–7.19 (m, 4 H), 7.16–7.09 (m, 1 H), 4.04–3.92 (m, 1 H), 3.09 (dd, J = 13.1, 4.0 Hz, 1 H), 2.89 (app t, J = 10.9 Hz, 1 H), 0.51–0.43 (m, 1 H), 0.42–0.35 (m, 1 H). ¹³C NMR (100 MHz, CD₂Cl₂) δ (ppm): 152.7, 149.9, 149.4, 146.9, 144.1, 140.1, 137.2, 136.5, 131.9 (q, J = 32.3 Hz), 129.9 (2 C), 129.2, 128.7, 128.4 (2 C), 128.1 (2 C), 127.4, 126.5, 126.3, 126.1, 125.8 (q, J = 3.0 Hz, 2 C), 125.8 (q, J = 271.3 Hz), 125.4, 67.2, 46.5, -5.7. ¹⁹F NMR (367 MHz, CD₂Cl₂) δ (ppm): -63.0.

(Phen)Ni{N(trifluoromethylsulfonyl)CH(Bn)CH₂} (Compound 11)

The title compound was prepared according to General Procedure 2 employing 2-benzyl-1-((trifluoromethyl)sulfonyl)aziridine (159 mg, 0.60 mmol) yielding the desired product as a red solid (214 mg, 85%) with minor THF impurities. ¹H NMR (400 MHz, CD₂Cl₂) δ (ppm): 9.48 (d, J = 4.0 Hz, 1 H), 8.43 (dd, J = 14.6, 8.1 Hz, 2 H), 8.28 (d, J = 4.9 Hz, 1 H), 7.96–7.81 (m, 3 H), 7.62–7.55 (m, 1 H), 7.33–7.20 (m, 4 H), 7.19–7.12 (m, 1 H), 4.43–4.33 (m, 1 H), 3.14 (d, J = 10.0 Hz, 1 H), 2.97 (app t, J = 11.5 Hz, 1 H), 0.98 – 0.91 (m, 1 H), 0.61 – 0.54 (s, 1 H). ¹³C NMR (100 MHz, CD₂Cl₂) δ (ppm): 152.4, 149.9, 147.1, 144.1, 139.6, 137.4, 136.9, 130.2 (2 C), 129.4, 128.8, 128.6 (2 C), 127.5, 126.6, 126.4 (2 C), 125.5, 122.3 (q, J = 328.8 Hz), 69.3, 47.0, -3.3. ¹⁹F NMR (367 MHz, CD₂Cl₂) δ (ppm): -75.2.
(Phen)Ni{N(Ts)CH(Bn)CH$_{13}$CO} and (S)-(Phen)Ni{N(Ts)CH(Bn)CH$_{13}$CO} (Compounds 12 and 12‘)

The title compounds were prepared according to General Procedure 3 employing 1 or 1’ (263 mg, 0.50 mmol) yielding the desired products as bright red solids (12, 250 mg, 93%) and (12’, 242 mg, 87%) with minor dichloroethane impurities. $^1$H NMR (400 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 9.58 (dd, $J = 5.2$, 1.0 Hz, 1 H), 9.32 (d, $J = 4.6$ Hz, 1 H), 8.46 (app. t, $J = 6.6$ Hz, 2 H), 7.99 (d, $J = 8.1$ Hz, 2 H), 7.97–7.89 (m, 2 H), 7.87–7.81 (m, 2 H), 7.19 (d, $J = 7.9$ Hz, 4 H), 7.07–6.97 (m, 3 H), 3.44–3.34 (m, 1 H), 3.19–2.08 (m, 2 H), 2.38 (s, 3 H), 2.26 (dd, $J = 16.2$, 5.0 Hz, 1 H), 2.04–1.93 (m, 1 H). $^{13}$C NMR (126 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 240.2 ($^{13}$C-enriched), 153.3, 151.5, 147.3, 144.6, 142.7, 141.3, 139.9, 137.9, 137.7, 130.7 (2 C), 129.4 (2 C), 129.2, 128.5, 128.4 (2 C), 127.7 (2 C), 126.9, 126.6, 126.3, 125.2, 125.2, 59.6 (d, $J = 20.0$ Hz), 46.7, 30.2, 21.6.

(Phen)Ni{N(Ts)CH(5-methylbenzo[1,3]dioxolyl)CH$_{13}$CO} (Compound 13)

The title compound was prepared according to General Procedure 3 employing 2 (285 mg, 0.50 mmol) yielding the desired product as a dark red solid (225 mg, 75%) with minor impurities. $^1$H NMR (400 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 9.53 (d, $J = 4.6$ Hz, 1 H), 9.40 (d, $J = 4.5$ Hz, 1 H), 8.47 (d, $J = 7.1$ Hz, 2 H), 8.04–7.80 (m, 6 H), 7.21 (d, $J = 7.4$ Hz, 2 H), 6.70 (s, 1 H), 6.62 (d, $J = 8.7$ Hz, 1 H), 6.43 (d, $J = 7.7$ Hz, 1 H), 5.66 (s, 1 H), 5.54 (s, 1 H), 3.23 (dd, $J = 12.7$, 5.7 Hz, 1 H), 3.12–2.99 (m, 2 H), 2.39 (s, 3 H), 2.26 (dd, $J = 16.2$, 4.5 Hz, 1 H), 2.01–1.92 (m, 1 H). $^{13}$C NMR (126 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 240.3 ($^{13}$C-enriched), 153.3, 151.5, 147.6, 147.2, 146.2, 144.6, 142.6, 141.4, 137.9, 137.7, 136.5, 133.4, 129.4 (2 C), 129.2, 127.7 (2 C), 127.0, 126.6, 126.3, 125.2, 123.7, 111.4, 108.0, 101.2, 59.4 (d, $J = 20.4$ Hz), 46.0, 30.2, 21.7.

(Phen)Ni{N(Ts)CH(Ph)CH$_{13}$CO} (Compound 14)

The title compound was prepared according to General Procedure 3 employing 3 (256 mg, 0.50 mmol) yielding the desired product as a bright red solid (224 mg, 83%) with minor dichloroethane impurities. $^1$H NMR (400 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 9.51–9.46 (m, 1 H), 9.41–9.36 (m, 1 H), 8.45–8.34 (m, 2 H), 8.08 (d, $J = 8.0$ Hz, 2 H), 7.90–7.80 (m, 3 H), 7.75–
7.67 (m, 1 H), 7.23 (d, J = 7.9 Hz, 2 H), 7.15–7.06 (m, 3 H), 7.00 (d, J = 7.4 Hz, 2 H), 3.28–3.20 (m, 1 H), 3.19–3.04 (m, 2 H), 2.36 (s, 3 H). $^{13}$C NMR (126 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 238.6 ($^{13}$C-enriched), 142.3, 141.7, 139.6, 139.5, 129.5 (2 C), 129.0, 129.0, 128.7 (2 C), 127.9 (2 C), 126.9, 125.3 (2 C), 71.6 (d, J = 17.6 Hz), 51.2 (d, J = 2.2 Hz), 21.7. Some aromatic carbon signals are missing.

(Phen)Ni{N(Ts)CH(methyl)CH$_2^{13}$CO} (Compound 15)

The title compound was prepared according to General Procedure 3 employing 4 (225 mg, 0.50 mmol) yielding the desired product as a bright red solid (193 mg, 81%) with dichloroethane impurities. $^1$H NMR (400 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 9.63 (d, J = 4.9 Hz, 1 H), 9.54 (d, J = 3.9 Hz, 1 H), 8.51–8.44 (m, 2 H), 8.11 (d, J = 7.7 Hz, 2 H), 7.98–7.82 (m, 4 H), 7.24 (d, J = 7.7 Hz, 2 H), 2.96–2.85 (m, 1 H), 2.39 (s, 3 H), 2.11 (dd, J = 15.9, 4.2 Hz, 1 H), 2.00–1.89 (m, 1 H), 1.55 (d, J = 6.5 Hz, 3 H). $^{13}$C NMR (126 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 240.2 ($^{13}$C-enriched), 142.8, 141.3, 129.3, 127.7, 125.3, 61.7 (d, J = 19.8 Hz), 48.5, 26.9, 21.6. Some aromatic carbon peaks are missing.

(Phen)Ni{N(Ts)CH(isopropyl)CH$_2^{13}$CO} (Compound 16)

The title compound was prepared according to General Procedure 3 employing 5 (239 mg, 0.50 mmol) yielding the desired product as a bright red solid (209 g, 82%) with minor dichloroethane impurities. $^1$H NMR (400 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 9.65 (d, J = 5.3 Hz, 1 H), 9.62 (d, J = 4.8 Hz, 1 H), 8.47 (d, J = 8.1 Hz, 2 H), 8.12 (d, J = 8.1 Hz, 2 H), 7.97–7.88 (m, 3 H), 7.85 (dd, J = 8.1, 5.3 Hz, 1 H), 7.23 (d, J = 8.1 Hz, 2 H), 2.57 (app q, J = 7.9 Hz, 1 H), 2.39 (s, 3 H), 2.30 (ddd, J = 16.4, 5.1, 1.7 Hz, 1 H), 2.13 (h, J = 6.8 Hz, 1 H), 1.77 (ddd, J = 16.4, 7.9, 6.5 Hz, 1 H), 1.24 (d, J = 6.7 Hz, 3 H), 1.12 (d, J = 6.7 Hz, 3 H). $^{13}$C NMR (126 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 240.4 ($^{13}$C-enriched), 152.9, 151.6, 147.2, 144.5, 142.8, 141.2, 137.9, 137.7, 129.2 (2 C), 129.2, 128.4, 127.9 (2 C), 126.9, 126.7, 125.3, 125.2, 58.2, 57.5 (d, J = 20.2), 35.8, 21.6, 20.2, 19.8.
(Phen)Ni{N(Ts)CH(methylene cyclohexyl)CH$_{13}$CO} (Compound 17)

The title compound was prepared according to General Procedure 3 employing 6 (266 mg, 0.50 mmol) yielding the desired product as a bright red solid (251 mg, 90%) with minor dichloroethane impurities.  

$^1$H NMR (400 MHz, CD$_2$Cl$_2$) δ (ppm): 9.65 (d, J = 5.4 Hz, 1 H), 9.56 (d, J = 4.9 Hz, 1 H), 8.47 (d, J = 8.0 Hz, 2 H), 7.97–7.89 (m, 3 H), 7.85 (dd, J = 8.1, 5.3 Hz, 1 H), 7.24 (d, J = 7.8 Hz, 2 H), 2.87–2.77 (m, 1 H), 2.20 (ddd, J = 13.7, 9.1, 4.7 Hz, 1 H), 2.07–1.92 (m, 2 H), 1.84–1.76 (m, 1 H), 1.70–1.53 (m, 5 H), 1.52–1.43 (m, 1 H), 1.31–1.11 (m, 5 H), 0.98–0.76 (m, 3 H).  

$^{13}$C NMR (126 MHz, CD$_2$Cl$_2$) δ (ppm): 240.7 (13C-enriched), 152.6, 151.5, 147.2, 144.5, 143.0, 141.3, 138.0, 137.8, 129.3 (2 C), 129.2, 128.5, 127.7 (2 C), 126.9, 126.7, 125.3, 125.2, 60.8 (d, J = 19.9 Hz), 50.1, 49.4, 34.8, 34.6, 33.4, 27.2, 27.1, 26.8, 21.6

(Phen)Ni{N(Ts)CH(3-(2-isopropyl-5-methyl cyclohexyl)methoxy)CH$_{13}$CO} (Compound 18)

The title compound was prepared according to General Procedure 3 employing 7 (302 mg, 0.50 mmol) yielding the desired product as a bright red solid in a mixture of diastereomers (1:1) (282 mg, 89%).  

$^1$H NMR (400 MHz, CD$_2$Cl$_2$) δ (ppm): 9.69 (app t, J = 4.7 Hz, 2 H), 9.58 (app t, J = 5.0 Hz, 2 H), 8.50 (dd, J = 7.7, 4.2 Hz, 4 H), 8.15 (app t, J = 7.1 Hz, 4 H), 8.02–7.91 (m, 6 H), 7.91–7.87 (m, 2 H), 7.28 (d, J = 7.7 Hz, 4 H), 4.11 (d, J = 8.8, 6.6 Hz, 1 H), 4.01 (dd, J = 8.6, 4.5 Hz, 1 H), 3.82–3.75 (m, 1 H), 3.65 (dd, J = 8.8, 3.9 Hz, 1 H), 3.12–2.95 (m, 4 H), 2.43 (s, 6 H), 2.31–2.06 (m, 4 H), 1.86 (dt, J = 14.9, 6.7 Hz, 2 H), 1.64–1.38 (m, 4 H), 1.35–1.22 (m, 2 H), 1.14–1.05 (m, 1 H), 0.91–0.74 (m, 12 H), 0.77 (d, J = 6.3 Hz, 3 H), 0.58–0.38 (m, 12 H).  

$^{13}$C NMR (126 MHz, CD$_2$Cl$_2$) δ (ppm): 240.3 (13C-enriched), 239.6 (13C-enriched), 152.9 (2 C), 151.4 (2 C), 147.2 (2 C), 144.4 (2 C), 142.7, 142.6, 141.4, 141.3, 137.8, 137.7, 137.6, 137.5, 129.3 (2 C), 129.3 (2 C), 129.2 (2 C), 128.4, 128.4, 127.9 (2 C), 127.8 (2 C), 126.8, 126.8, 126.7 (2 C), 125.3, 125.2, 125.2 (2 C), 79.7, 79.2, 74.8, 74.5, 57.2 (d, J = 20.1 Hz), 56.9 (d, J = 19.9 Hz), 52.9, 52.6, 49.0, 48.9, 40.8, 40.7, 35.0 (2 C), 31.9, 31.8, 25.7, 25.7, 23.4 (2 C), 22.6, 22.5, 21.6 (2 C), 21.0, 20.8, 16.2, 15.9.
(Phen)Ni{N(4-methoxybenzenesulfonyl)CH(Bn)CH_{13}CO} (Compound 19)

The title compound was prepared according to General Procedure 3 employing 8 (271 mg, 0.50 mmol) yielding the desired product as a dark red solid (181 mg, 63%) with minor THF impurities. $^1$H NMR (400 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 9.58 (dd, $J = 5.3$, 1.4 Hz, 1 H), 9.32 (d, $J = 4.1$ Hz, 1 H), 8.46 (d, $J = 8.0$, 6.4, 1.5 Hz, 2 H), 8.0 (d, $J = 8.8$ Hz, 2 H), 7.85 (app q, $J = 8.8$ Hz, 2 H), 7.84 (dt, $J = 8.1$, 5.1 Hz, 2 H), 7.19 (d, $J = 6.8$ Hz, 2 H), 7.07–6.98 (m, 3 H), 6.78 (d, $J = 8.8$ Hz, 2 H), 3.84 (s, 3 H), 3.44–3.36 (m, 1 H), 3.17–2.07 (m, 2 H), 2.27 (ddd, $J = 16.7$, 5.2, 1.3 Hz, 1 H), 2.03 (dt, $J = 16.1$, 6.6 Hz, 1 H). $^{13}$C NMR (126 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 240.4 ($^{13}$C-enriched), 153.2, 151.5, 147.2, 144.5, 139.9, 137.9, 137.7, 137.6, 130.6 (2 C), 129.5, 129.2, 128.4 (2 C), 128.3 (2 C), 126.9, 126.6, 126.3, 125.3, 125.2, 125.1, 113.8 (2 C), 68.3, 59.7 (d, $J = 20.1$ Hz), 55.9, 46.7.

(Phen)Ni{N(4-trifluoromethylbenzenesulfonyl)CH(Bn)CH_{13}CO} (Compound 20)

The title compound was prepared according to General Procedure 3 employing 10 (290 mg, 0.50 mmol) yielding the desired product as a bright red solid (294 mg, 97%) with minor dichloroethane impurities. $^1$H NMR (400 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 9.56 (d, $J = 5.3$ Hz, 1 H), 9.23 (d, $J = 5.0$ Hz, 1 H), 8.48 (dd, $J = 8.0$, 4.9 Hz, 2 H), 8.20 (d, $J = 8.1$ Hz, 2 H), 7.94 (app q, $J = 8.8$ Hz, 2 H), 7.86 (dd, $J = 8.1$, 5.4 Hz, 1 H), 7.83 (dd, $J = 8.2$, 5.0 Hz, 1 H), 7.63 (d, $J = 8.2$ Hz, 2 H), 7.18 (d, $J = 7.2$ Hz, 2 H), 7.07–7.01 (m, 3 H), 3.48–3.40 (m, 1 H), 3.18–3.09 (m, 2 H), 2.33 (dd, $J = 16.3$, 4.9 Hz, 1 H), 2.12–2.02 (m, 1 H). $^{13}$C NMR (126 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 239.1 ($^{13}$C-enriched), 171.1, 152.9, 151.4, 149.0, 147.1, 144.4, 139.6, 138.1, 137.9, 132.2 (q, $J = 32.5$ Hz), 130.5, 129.7, 129.2, 128.5, 128.4 (2 C), 128.1 (2 C), 126.9, 126.7, 126.4, 125.9 (q, $J = 3.6$ Hz), 125.3, 125.1, 124.4 (q, $J = 272.5$ Hz), 59.6 (d, $J = 19.7$ Hz), 54.4, 46.6. $^{19}$F NMR (367 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): -63.0.
(Phen)Ni{N(trifluoromethylsulfonyl)CH(Bn)CH$_{13}$CO} (Compound 21)

The title compound was prepared according to General Procedure 3 employing 11 (252 mg, 0.50 mmol) yielding the desired product as a dark red solid (219 mg, 82%) with minor impurities. $^1$H NMR (400 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 9.51 (d, $J = 5.4$ Hz, 1 H), 9.03 (d, $J = 4.0$ Hz, 1 H), 8.49–8.43 (m, 2 H), 7.92 (d, $J = 4.7$ Hz, 2 H), 7.90–7.83 (m, 1 H), 7.82–7.76 (m, 1 H), 7.29 (d, $J = 7.3$ Hz, 2 H), 7.14 (app t, $J = 7.4$ Hz, 2 H), 7.10–7.04 (m, 1 H), 3.54–3.47 (m, 2 H), 3.37–3.29 (m, 1 H), 2.60–2.51 (m, 2 H). $^{13}$C NMR (126 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 236.1 ($^{13}$C-enriched), 152.4, 151.3, 146.8, 144.1, 138.8, 138.3, 138.2, 130.5 (2 C), 129.1, 128.5 (2 C), 128.5, 127.0, 126.8, 126.7, 125.3, 125.1, 121.5 (q, $J = 327.7$ Hz), 58.4 (d, $J = 19.4$ Hz), 55.1 (d, $J = 0.8$ Hz), 46.9. $^{19}$F NMR (367 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): -75.9.

(Phen)Ni{N(methylsulfonyl)CH(Bn)CH$_{13}$CO} (Compound 22)

The title compound was prepared according to General Procedure 3 employing 9 (235 mg, 0.50 mmol) yielding the desired product as a bright red solid (211 mg, 88%) with minor dichloroethane impurities. $^1$H NMR (400 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 9.60 (d, $J = 5.1$ Hz, 1 H), 9.06 (d, $J = 5.1$ Hz, 1 H), 8.48–8.42 (m, 2 H), 7.95–7.88 (m, 2 H), 7.85 (dd, $J = 8.0$, 5.4 Hz, 1 H), 7.80 (dd, $J = 8.2$, 4.9 Hz, 1 H), 7.30 (d, $J = 7.3$ Hz, 2 H), 7.21–7.14 (m, 2 H), 7.14–7.07 (m, 1 H), 3.45–3.36 (m, 1 H), 3.16–3.07 (m, 2 H), 2.83–2.74 (m, 1 H), 2.63 (s, 3 H), 2.57 (dd, $J = 16.0$, 4.9 Hz, 2 H). $^{13}$C NMR (126 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 240.2 ($^{13}$C-enriched), 152.5, 151.5, 147.1, 144.4, 140.5, 137.9, 137.8, 130.6 (2 C), 129.1, 128.4 (2 C), 128.4, 126.8, 126.7, 126.5, 125.2, 125.0, 62.2 (d, $J = 20.1$ Hz), 55.4 (d, $J = 1.2$ Hz), 46.6, 41.3.

Methyl 3-((4-methylphenyl)sulfonamide-4-phenylbutanoate-1-$^{13}$C and (S)-methyl 3-((4-methylphenyl)sulfonamide-4-phenylbutanoate-1-$^{13}$C (Compounds 23 and 23’)

Hydrolysis-Methylation (Scheme 4): The title compound was prepared according to General Procedure 4 employing 12 or 12’ (111 mg, 0.20 mmol). Flash column chromatography (10-15% EtOAc in pentane) afforded the products as colorless solids (23, 48 mg, 69%) and (23’, 43 mg, 61%). Methoxide ring-opening (Scheme 5): In an argon filled glovebox to a 10 mL flask was added 12 (28 mg, 0.050
mmol, 1.0 equiv) and NaOMe (5 mg, 0.1 mmol, 2.0 equiv) which were dissolved in DMA (1 mL). The reaction was sealed with a screw cap fitted with a Teflon® seal, removed from the glovebox and stirred at room temperature for 16 h. The reaction was quenched with sat. NH₄Cl (aq) and extracted with Et₂O. The combined organic phases were dried over Na₂SO₄, filtered and concentrated in vacuo. Flash column chromatography (10% EtOAc in pentane) afforded the product as a colorless solid (9 mg, 51%). The physical and spectral properties were in accordance with the literature.¹⁹ Optical rotation for 23': [α]²³_D = -17.0 (c = 1.0, CH₂Cl₂), Lit: [α]¹⁹_D = -18.1 (c = 0.6, CH₂Cl₂). mp for 23: 100–103 °C, mp(lit): 100–103 °C.

**Methyl 3-((4-methylphenylsulfonamide)butanoate-1-¹³C (Compound 24)**

The title compound was prepared according to General Procedure 4 employing 15 (96 mg, 0.20 mmol). Flash column chromatography (5-15% EtOAc in pentane) afforded the product as a colorless solid (34 mg, 62%). mp: 57–58 °C.

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.61 (d, J = 8.3 Hz, 2 H), 7.23–7.17 (m, 5 H), 7.00 (dd, J = 6.6, 2.6 Hz, 2 H), 5.23 (d, J = 8.4 Hz, 1 H), 3.82–3.70 (m, 1 H), 3.61 (d, J = 3.9 Hz, 3 H), 2.86–2.69 (m, 2 H), 2.50–2.43 (m, 2 H), 2.40 (s, 3 H). ¹³C NMR (100 MHz, CDCl₃) δ (ppm): 171.8 (¹³C-enriched), 143.3, 137.6, 136.9, 129.7 (2 C), 129.4 (2 C), 128.7 (2 C), 127.1 (2 C), 126.9, 52.0 (d, J = 0.8 Hz), 51.8 (d, J = 2.7 Hz), 40.9 (d, J = 1.2 Hz), 38.1 (d, J = 57.5 Hz) 21.6. HRMS C₁₇¹³CH₂₁NO₄S [M+H⁺]; calculated 349.1298, found 349.1304.

**Methyl 4-methyl-3-((4-methylphenylsulfonamide)pentanoate-1-¹³C (Compound 25)**

The title compound was prepared according to General Procedure 4 employing 16 (101 mg, 0.20 mmol). Flash column chromatography (10% EtOAc in pentane) afforded the product as a colorless solid (49 mg, 81%). mp: 88–90 °C.

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.75 (d, J = 8.2 Hz, 2 H), 7.29 (d, J = 8.1 Hz, 2 H), 5.21 (d, J = 8.5 Hz, 1 H), 3.73–3.63 (m, 1 H), 3.61 (d, J = 3.9 Hz, 3 H), 2.45–2.40 (m, 5 H), 1.13 (d, J = 6.7 Hz, 3 H). ¹³C NMR (100 MHz, CDCl₃) δ (ppm): 171.7 (¹³C-enriched), 143.5, 138.2, 129.8 (2 C), 127.2 (2 C), 51.8 (d, J = 2.9 Hz), 46.7 (d, J = 1.6 Hz), 40.7 (d, J = 57.5 Hz), 21.6, 21.2 (d, J = 1.7 Hz). HRMS C₁₁¹³CH₁⁷NO₄S [M+H⁺]; calculated 273.0985, found 273.0973.
$^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 7.76 (d, J = 8.3 Hz, 2 H), 7.30 (d, J = 8.0 Hz, 2 H), 5.03 (d, J = 9.1 Hz, 1 H), 3.63 (d, J = 3.9 Hz, 3 H), 3.62–3.54 (m, 1 H), 2.46–2.43 (m, 2 H), 2.42 (s, 3 H), 1.66–1.56 (m, 2 H), 1.54–1.46 (m, 2 H), 1.44–1.32 (m, 2 H), 1.27–0.87 (m, 5 H), 0.80 (qd, J = 12.2, 3.5 Hz, 1 H), 0.65 (qd, J = 12.6, 3.3 Hz, 1 H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm): 172.0 ($^{13}$C-enriched), 143.5, 138.0, 137.9, 129.8 (2 C), 129.8 (2 C), 127.2 (2 C), 127.2 (2 C), 79.6, 79.5, 68.7 (d, J = 1.0 Hz), 68.5 (d, J = 1.0 Hz), 51.8, 51.7, 50.7, 48.2 (d, J = 1.3 Hz), 42.7, 39.3 (d, J = 57.3 Hz), 34.0, 33.7, 32.6, 26.5, 26.3, 26.0, 21.0. HRMS C$_{17}$H$_{27}$NO$_4$S [M+H$^+$]; calculated 355.1767, found 355.1764.

**Methyl 4-cyclohexyl-3-((4-methylphenylsulfonamide)butanoate-1-$^{13}$C (Compound 26)**

The title compound was prepared according to General Procedure 4 employing 17 (112 mg, 0.20 mmol). Flash column chromatography (10% EtOAc in pentane) afforded the product as a colorless solid (53 mg, 74%). mp: 110–112 °C. $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 7.76 (d, J = 8.3 Hz, 2 H), 7.30 (d, J = 8.0 Hz, 2 H), 5.03 (d, J = 9.1 Hz, 1 H), 3.63 (d, J = 3.9 Hz, 3 H), 3.62–3.54 (m, 1 H), 2.46–2.43 (m, 2 H), 2.42 (s, 3 H), 1.66–1.56 (m, 2 H), 1.54–1.46 (m, 2 H), 1.44–1.32 (m, 2 H), 1.27–0.87 (m, 5 H), 0.80 (qd, J = 12.2, 3.5 Hz, 1 H), 0.65 (qd, J = 12.6, 3.3 Hz, 1 H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm): 172.0 ($^{13}$C-enriched), 143.5, 138.2, 129.8 (2 C), 127.3 (2 C), 51.8 (d, J = 2.5 Hz), 48.2 (d, J = 1.3 Hz), 42.7, 39.3 (d, J = 57.3 Hz), 34.0, 33.7, 32.6, 26.5, 26.3, 26.0, 21.0. HRMS C$_{17}$H$_{27}$NO$_4$S [M+H$^+$]; calculated 355.1767, found 355.1764.

**Methyl 4-(((1$^R$, 2$^S$, 5$^R$)-2-isopropyl-5-methylcyclohexyloxy)-3-((4-methylphenyl)sulfonamide)butanoate-1-$^{13}$C (Compound 27)**

The title compound was prepared according to General Procedure 4 employing 18 (127 mg, 0.20 mmol). Flash column chromatography (6% EtOAc in pentane) afforded the product as a colorless oil (70 mg, 82%). The product was isolated as a mixture of diastereomers (1:1). $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 7.75 (dd, J = 8.2, 1.7 Hz, 4 H), 7.30 (d, J = 7.6 Hz, 4 H), 5.24 (d, J = 8.6 Hz, 1 H), 5.17 (d, J = 8.7 Hz, 1 H), 3.80–3.66 (m, 2 H), 3.60 (d, J = 3.9 Hz, 6 H), 3.55 (dd, J = 9.1, 4.0 Hz, 1 H), 3.43 (dd, J = 9.3, 4.9 Hz, 1 H), 3.20 (dd, J = 9.3, 3.5 Hz, 1 H), 3.14 (dd, J = 9.1, 5.8 Hz, 1 H), 2.96–2.87 (m, 2 H), 2.68–2.58 (m, 2 H), 2.57–2.46 (m, 2 H), 2.46 (s, 6 H), 2.06–1.95 (m, 2 H), 1.95–1.88 (m, 1 H), 1.80 (m, 1 H), 1.66–1.56 (m, 4 H), 1.17–1.07 (m, 2 H), 0.90–0.84 (m, 15 H), 0.63 (dd, J = 6.0, 6.8 Hz, 7 H). $^{13}$C NMR (100 MHz, CD$_2$Cl$_3$) δ (ppm): 171.7 ($^{13}$C-enriched), 171.6 ($^{13}$C-enriched), 143.5 (2 C), 138.0, 137.9, 129.8 (2 C), 129.8 (2 C), 127.2 (2 C), 127.2 (2 C), 79.6, 79.5, 68.7 (d, J = 1.0 Hz), 68.5 (d, J = 1.0 Hz), 51.8, 51.7, 50.7.
50.7, 48.3, 48.2, 40.1, 40.0, 36.7 (d, J = 57.7 Hz), 36.4 (d, J = 57.7 Hz), 34.5 (2 C), 31.5, 31.5, 25.7 (2 C), 23.3, 23.2, 22.4 (2 C), 21.6 (2 C), 21.0, 21.0, 16.2, 16.1. HRMS C_{21}{^{13}}CH_{33}NO_5S [M+H^+]; calculated 427.2342, found 427.2341.

**Methyl 4-(benzo[d][1,3]dioxol-5-yl)-3-((4-methylphenylsulfonamide)butanoate-1-{^{13}}C**

(Compound 28)

The title compound was prepared according to General Procedure 4 employing 13 (108 mg, 0.20 mmol). Flash column chromatography (16% EtOAc in pentane) afforded the product as a colorless oil (21 mg, 26%). \(^1^H \text{NMR (400 MHz, CDCl}_3) \)\( \delta \) (ppm): 7.60 (d, J = 8.2 Hz, 2 H), 7.22 (d, J = 8.0 Hz, 2 H), 6.63 (d, J = 7.8 Hz, 1 H), 6.47–6.41 (m, 2 H), 5.91 (d, J = 3.2 Hz, 2 H), 5.10 (d, J = 8.2 Hz, 1 H), 3.71–3.64 (m, 1 H), 3.63 (d, J = 3.8 Hz, 3 H), 2.69 (qd, J = 13.9, 7.1 Hz, 2 H), 2.52–2.46 (m, 2 H), 2.41 (s, 3 H). \(^{13}C \text{NMR (100 MHz, CDCl}_3) \)\( \delta \) (ppm): 171.8 (\(^{13}C\)-enriched), 147.9, 146.6, 143.4, 137.5, 130.6, 129.6 (2 C), 127.2 (2 C), 122.5, 109.5, 108.4, 101.1, 52.2 (d, J = 1.0 Hz), 51.9 (d, J = 2.7 Hz), 40.5, 38.1 (d, J = 57.4 Hz), 21.6. HRMS C_{18}{^{13}}CH_{21}NO_6S [M+H^+]; calculated 393.1196, found 393.1204.

**Methyl 3-((4-methylphenyl)sulfonamide-2-phenylpropionate-1-{^{13}}C**

(Compound 29)

The title compound was prepared according to General Procedure 4 employing 14 (108 mg, 0.20 mmol). Flash column chromatography afforded the product as a light yellow oil (45 mg, 67%). \(^1^H \text{NMR (400 MHz, CDCl}_3) \)\( \delta \) (ppm): 7.70 (d, J = 8.2 Hz, 2 H), 7.33–7.26 (m, 5 H), 7.17–7.12 (m, 2 H), 4.98 (t, J = 6.7 Hz, 1 H), 3.89–3.80 (m, 1 H), 3.65 (d, J = 3.9 Hz, 3 H), 3.50–3.40 (m, 1 H), 3.30–3.29 (m, 1 H), 2.42 (s, 3 H). \(^{13}C \text{NMR (100 MHz, CDCl}_3) \)\( \delta \) (ppm): 173.0 (\(^{13}C\)-enriched), 143.6, 137.1, 135.7 (d, J = 2.6 Hz), 129.9 (2 C), 129.2 (2 C), 128.1, 128.1 (d, J = 2.2 Hz, 2 C), 127.1 (2 C), 52.5 (d, J = 2.8 Hz), 51.8 (d, J = 57.3 Hz), 45.8, 21.6. HRMS C_{16}{^{13}}CH_{19}NO_4S [M+H^+]; calculated 335.1141, found 335.1141.
Methyl 4-phenyl-3-((4-(trifluoromethyl)phenyl)sulfonamido)butanoate-1-\(^{13}\)C (30)

The title compound was prepared according to General Procedure 4 employing 20 (122 mg, 0.20 mmol). Flash column chromatography (10-15% EtOAc in pentane) afforded the product as a colorless solid (52 mg, 65%). mp: 134–136 °C. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.73 (d, \(J = 8.2\) Hz, 2 H), 7.60 (d, \(J = 8.4\) Hz, 2 H), 7.18–7.11 (m, 3 H), 6.98–6.93 (m, 2 H), 5.24 (d, \(J = 8.6\) Hz, 1 H), 3.81–3.73 (m, 1 H), 3.66 (d, \(J = 3.9\) Hz, 3 H), 2.84 (dd, \(J = 13.8, 6.3\) Hz, 1 H), 2.76 (dd, \(J = 13.8, 8.1\) Hz, 1 H), 2.60 (dd, \(J = 7.1, 5.4\) Hz, 2 H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) (ppm): 171.7 (\(^{13}\)C-enriched), 143.9, 136.8, 134.0 (q, \(J = 32.9\) Hz), 129.2 (2 C), 128.8 (2 C), 127.4 (2 C), 127.1, 126.2 (q, \(J = 3.6\) Hz, 2 C), 120.7 (q, \(J = 272.9\) Hz), 52.7 (0.7 Hz), 52.0 (d, \(J = 2.7\) Hz), 40.9, 39.0 (d, \(J = 57.5\) Hz). \(^{19}\)F NMR (367 MHz, CDCl\(_3\)) \(\delta\) (ppm): -63.2. HRMS C\(_{17}\)F\(_3\)NO\(_4\)S [M+H\(^+\)]; calculated 403.1015, found 403.1005.

Methyl 3-((4-methoxyphenyl)sulfonamido)-4-phenylbutanoate-1-\(^{13}\)C (Compound 31)

The title compound was prepared according to General Procedure 4 employing 19 (114 mg, 0.20 mmol). Flash column chromatography (10-20% EtOAc in pentane) afforded the product as a yellow oil (40 mg, 55%). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.65 (d, \(J = 8.9\) Hz, 2 H), 7.23–7.17 (m, 3 H), 7.02–6.98 (m, 2 H), 6.88 (d, \(J = 8.9\) Hz, 2 H), 5.06 (d, \(J = 8.4\) Hz, 1 H), 3.86 (s, 3 H), 3.77–3.67 (m, 1 H), 3.63 (d, \(J = 3.9\) Hz, 3 H), 2.83–2.76 (m, 2 H), 2.48 (d, \(J = 18.2\) Hz, 2 H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) (ppm): 171.9 (\(^{13}\)C-enriched), 162.9, 136.9, 132.2, 129.4 (2 C), 129.3 (2 C), 128.8 (2 C), 127.0, 114.2 (2 C), 55.6, 51.8 (d, \(J = 1.1\) Hz), 40.8 (d, \(J = 1.7\) Hz), 38.1, 37.6. HRMS C\(_{17}\)F\(_2\)NO\(_5\)S [M+H\(^+\)]; calculated 365.1247, found 365.1230.
Methyl 3-(methylsulfonamido)-4-phenylbutanoate-1\(^{13}\)C (Compound 32)

The title compound was prepared according to General Procedure 4 employing 22 (96 mg, 0.20 mmol). Flash column chromatography (10-20% EtOAc in pentane) afforded the product as a yellow oil (23 mg, 42%). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.36–7.31 (m, 2 H), 7.28–7.21 (m, 3 H), 5.00 (d, \(J = 9.4\) Hz, 1 H), 3.99–3.90 (m, 1 H), 3.72 (d, \(J = 3.9\) Hz, 3 H), 2.91 (d, \(J = 7.1\) Hz, 2 H), 2.72–2.64 (m, 1 H), 2.58 (d, \(J = 21.3\) Hz, 1 H), 2.53 (s, 3 H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) (ppm): 171.8 (\(^{13}\)C enriched), 137.5, 129.7 (2 C), 128.9 (2 C), 127.2, 53.0 (d, \(J = 1.3\) Hz), 52.0 (d, \(J = 2.7\) Hz), 41.6 (d, \(J = 1.9\) Hz), 41.1, 39.4 (d, \(J = 57.7\) Hz). HRMS C\(_{11}\)\(^{13}\)CH\(_{17}\)NO\(_4\)S [M+H\(^+\)]; calculated 273.0985, found 273.0981.

Methyl 4-phenyl-3-((1,1,1-trifluoro-N-methylmethyl)sulfonamido)butanoate-1\(^{13}\)C (Compound 33)

The title compound was prepared according to General Procedure 4 employing 20 (107 mg, 0.20 mmol). N-methylation was observed due to the excess of TMSCH\(_2\)N\(_2\) employed (2.0 equiv) and the increased reactivity of the triflyl-substituted nitrogen. Flash column chromatography (10% EtOAc in pentane) afforded the product as a colorless oil (39 mg, 64%). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.34–7.28 (m, 2 H), 7.27–7.21 (m, 1 H), 7.17 (d, \(J = 7.1\) Hz, 2 H), 4.58–4.46 (m, 1 H), 3.62 (d, \(J = 3.9\) Hz, 3 H), 2.97 (s, 3 H), 2.96–2.83 (m, 2 H), 2.55 (td, \(J = 7.2, 3.2\) Hz, 2 H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) (ppm): 170.3 (\(^{13}\)C-enriched), 136.1, 129.3 (2 C), 129.0 (2 C), 127.5, 120.0 (q, \(J = 320.9\) Hz), 52.2 (d, \(J = 2.6\) Hz), 39.9 (d, \(J = 2.7\) Hz), 37.5, 36.9, 29.9. \(^{19}\)F NMR (367 MHz, CD\(_2\)Cl\(_2\)) \(\delta\) (ppm): -76.2. HRMS C\(_{12}\)\(^{13}\)CH\(_{16}\)F\(_3\)NO\(_4\)S\(^+\) [M+H\(^+\)]; calculated 341.0858, found 341.0855.

4-Methyl-N-(4-oxo-1-phenylheptan-2-yl-4\(^{13}\)C)benzenesulfonamide (Compound 34)

The title compound was prepared according to General Procedure 5 employing n-propylzinc bromide (0.5 M in THF, 0.30 mL, 0.15 mmol, 3.0 equiv). Flash column chromatography (10-15% EtOAc in pentane) afforded the product as a
colorless solid (12.4 mg, 69%). mp: 73–75 °C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ (ppm): 7.58 (d, $J = 8.3$ Hz, 2 H), 7.23–7.16 (m, 5 H), 6.99–6.92 (m, 2 H), 5.14 (d, $J = 8.3$ Hz, 1 H), 3.78–3.66 (m, 1 H), 2.81 (dd, $J = 13.6$, 7.5 Hz, 1 H), 2.72 (dd, $J = 13.7$, 7.1 Hz, 1 H), 2.66–2.49 (m, 2 H), 2.40 (s, 3 H), 2.23 (tdd, $J = 7.2$, 5.5, 1.9 Hz, 2 H), 1.51 (dtq, $J = 11.2$, 7.3, 3.7 Hz, 2 H), 0.87 (t, $J = 7.4$ Hz, 3 H). $^{13}$C-NMR (100 MHz, CDCl$_3$) $\delta$ (ppm): 210.1 ($^{13}$C-enriched), 143.3, 137.6, 137.4, 129.8 (2 C), 129.2 (2 C), 128.8 (2 C), 127.1 (2 C), 126.9, 51.8 (d, $J = 1.8$ Hz), 45.7 (d, $J = 4.7$ Hz), 45.3 (d, $J = 3.2$ Hz), 40.6, 21.6, 17.1 (d, $J = 1.8$ Hz), 13.8 (d, $J = 3.3$ Hz). HRMS C$_{19}$H$_{25}$NO$_3$S [M+H$^+$]; calculated 361.1661, found 361.1653.

$N$-(5-Cyclopentyl-4-oxo-1-phenylpentan-2-yl-4-$^{13}$C)-4-methylbenzenesulfonamide (Compound 35)

The title compound was prepared according to General Procedure 5 employing 5-hexenylzinc bromide (0.5 M in THF, 0.30 mL, 0.15 mmol, 3.0 equiv). The organozinc reagent had undergone a rearrangement reaction forming (cyclopentylmethyl)zinc bromide. Flash column chromatography (10–20% EtOAc in pentane) afforded the product as an amorphous solid (6.5 mg, 33%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ (ppm): 7.51 (d, $J = 7.0$ Hz, 2 H), 7.17–7.10 (m, 5 H), 6.92–6.86 (m, 2 H), 5.08 (d, $J = 8.0$ Hz, 1 H), 3.64 (sep, $J = 6.0$ Hz, 1 H), 2.75 (dd, $J = 7.6$, 13.6 Hz, 1 H), 2.65 (dd, $J = 7.2$, 13.6 Hz, 1 H), 2.53 (dt, $J = 4.8$, 18.4 Hz, 1 H), 2.46 (dt, $J = 5.6$, 18.0 Hz, 1 H), 2.33 (s, 3 H), 2.20 (dd, $J = 5.5$, 7.2 Hz, 2 H), 2.04 (quin, $J = 8.0$ Hz, 1 H), 1.75–1.64 (m, 2 H), 1.58–1.40 (m, 4 H), 1.01–0.87 (m, 2 H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ (ppm): 210.1 ($^{13}$C-enriched), 143.3, 137.6, 137.4, 129.8 (2 C), 129.2 (2 C), 128.8 (2 C), 127.1 (2 C), 126.9, 51.8 (d, $J = 1.9$ Hz), 49.9 (d, $J = 39.5$ Hz), 45.6 (d, $J = 37.8$ Hz), 40.6, 35.5 (d, $J = 1.9$ Hz), 32.7 (d, $J = 2.7$ Hz) 29.9, 25.1 (2 C), 21.7. HRMS C$_{22}$H$_{29}$NO$_3$S [M+H$^+$]; calculated 401.1974, found 401.1983.

Ethyl 9-(methylphenyl)sulfonamide)-7-oxo-10-phenyldecanoate-7-$^{13}$C (Compound 36)

The title compound was prepared according to General Procedure 5 employing 6-ethoxy-6-oxohexylzinc bromide (0.5 M in THF, 0.30 mL, 0.15 mmol, 3.0 equiv). Flash column chromatography (2–4% EtOAc in CH$_2$Cl$_2$) afforded the product as an amorphous solid (5.4 mg, 25%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ (ppm): 7.57 (d, $J = 8.0$ Hz, 2 H), 7.23–7.15 (m, 5 H), 6.97–6.93 (m, 2 H), 5.11 (d, $J = 8.0$ Hz, 2 H), 5.08 (d, $J = 8.3$ Hz, 1 H), 3.64 (sep, $J = 6.0$ Hz, 1 H), 2.75 (dd, $J = 7.6$, 13.6 Hz, 1 H), 2.65 (dd, $J = 7.2$, 13.6 Hz, 1 H), 2.53 (dt, $J = 4.8$, 18.4 Hz, 1 H), 2.46 (dt, $J = 5.6$, 18.0 Hz, 1 H), 2.33 (s, 3 H), 2.20 (dd, $J = 5.5$, 7.2 Hz, 2 H), 2.04 (quin, $J = 8.0$ Hz, 1 H), 1.75–1.64 (m, 2 H), 1.58–1.40 (m, 4 H), 1.01–0.87 (m, 2 H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ (ppm): 210.1 ($^{13}$C-enriched), 143.3, 137.6, 137.4, 129.8 (2 C), 129.2 (2 C), 128.8 (2 C), 127.1 (2 C), 126.9, 51.8 (d, $J = 1.9$ Hz), 49.9 (d, $J = 39.5$ Hz), 45.6 (d, $J = 37.8$ Hz), 40.6, 35.5 (d, $J = 1.9$ Hz), 32.7 (d, $J = 2.7$ Hz) 29.9, 25.1 (2 C), 21.7. HRMS C$_{22}$H$_{29}$NO$_3$S [M+H$^+$]; calculated 401.1974, found 401.1983.
Hz, 1 H), 4.12 (q, $J = 7.2$ Hz, 2 H), 3.71 (dq, $J = 7.4$, 12.9 Hz, 1 H), 2.80 (dd, $J = 7.6$, 13.6 Hz, 1 H), 2.71 (dd, $J = 7.2$, 14.0 Hz, 1 H), 2.61 (dt, $J = 4.8$, 18.0 Hz, 1 H), 2.54 (dt, $J = 5.6$, 18.0 Hz, 1 H), 2.40 (s, 3 H), 2.27 (quin, $J = 7.2$ Hz, 4 H), 1.60 (quin, $J = 7.6$ Hz, 2 H), 1.50 (dq, $J = 3.5$, 7.5 Hz, 2 H), 1.31-1.27 (m, 2 H), 1.25 (t, $J = 7.2$ Hz, 3 H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ (ppm): 209.8 ($^{13}$C-enriched), 173.7, 143.3, 137.6, 137.3, 129.8 (2 C), 129.2 (2 C), 128.8 (2 C), 127.1 (2 C), 126.9, 60.4, 51.8, 45.5 (d, $J = 38.2$ Hz), 43.3 (d, $J = 39.4$ Hz), 40.6, 34.2, 28.7 (d, $J = 3.2$ Hz), 24.8, 23.2 (d, $J = 1.8$ Hz), 21.6, 14.4. HRMS C$_{24}$H$_{33}$NO$_5$S [M+H$^+$]: calculated 461.2186, found 461.2192.

3-((4-Methylphenyl)sulfonamide)-N,4-diphenylbutanamide-1$^{13}$C (Compound 37)

In an argon filled glovebox in a vial was added KOrBu (16.8 mg, 0.15 mmol, 3.0 equiv) and aniline (9 $\mu$L, 0.10 mmol, 2.0 equiv) in DMA (0.5 mL). The solution stirred for 5-10 min at room temperature. The solution was taken up by syringe and dropwise added to a solution of complex 12 (28 mg, 0.05 mmol, 1.0 equiv) in DMA (0.5 mL) in a 10 mL flask. The reaction was sealed with a screw cap fitted with a Teflon ® seal and removed from the glovebox and stirred at room temperature for 16 h. The reaction was quenched with 2M HCl$_{aq}$ and extracted with dichloromethane. The combined organic phases were dried over Na$_2$SO$_4$, filtered and concentrated in vacuo. Flash column chromatography (2.5% EtOAc in CH$_2$Cl$_2$) afforded the product as a colorless oil (10.4 mg, 51%).

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ (ppm): 7.60 (d, $J = 8.4$ Hz, 2 H), 7.46 (d, $J = 8.0$ Hz, 2 H), 7.38 (bs, 1 H), 7.33 (t, $J = 8.0$ Hz, 2 H), 7.24-7.20 (m, 3 H), 7.18 (d, $J = 8.0$ Hz, 2 H), 7.13 (t, $J = 7.4$ Hz, 1 H), 7.02 (dd, $J = 3.6$, 7.2 Hz, 2 H), 5.47 (d, $J = 7.2$ Hz, 1 H), 3.75 (tt, $J = 3.6$, 10.2 Hz, 1 H), 2.94-2.83 (m, 2 H), 2.59 (dt, $J = 5.6$, 15.2 Hz, 1 H), 2.43 (t, $J = 5.2$ Hz, 1 H), 2.38 (s, 3 H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ (ppm): 168.8 ($^{13}$C-enriched), 143.6, 137.5, 137.1 (2 C), 129.8 (2 C), 129.3 (2 C), 129.2 (2 C), 128.9 (2 C), 127.2 (2 C), 127.0, 124.8, 120.2 (d, $J = 1.9$ Hz, 2 C), 52.7, 40.9, 40.6 (d, $J = 50.4$ Hz), 21.7. HRMS C$_{22}$H$_{33}$N$_2$O$_5$S [M+H$^+$]: calculated 410.1614, found 410.1610.

S-benzyl 3-((4-methylphenyl)sulfonamido)-4-phenylbutanethioate-1$^{13}$C (Compound 38)

In an argon filled glovebox in a 10 mL flask was added complex 12 (28 mg, 0.05 mmol, 1.0 equiv.) which was dissolved in dichloroethane (1 mL). Benzyl mercaptan (12 $\mu$L, 0.10 mmol, 2.0
equiv) and Et₃N (14 µL, 0.10 mmol, 2.0 equiv) were added and the reaction was then sealed with a screw cap fitted with a Teflon ® seal, removed from the glovebox and stirred at room temperature for 16 h. The reaction was quenched with 1M HCl(aq) and extracted with dichloromethane. The combined organic phases were dried over Na₂SO₄, filtered and concentrated in vacuo. Flash column chromatography (5-10% EtOAc in pentane) afforded the product as an off-white solid (13 mg, 0.12 mmol, 58%). mp: 77–79 °C. ¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.60 (d, J = 8.2 Hz, 2 H), 7.34–7.24 (m, 5 H), 7.22–7.17 (m, 5 H), 7.02–6.95 (m, 2 H), 5.01 (d, J = 8.2 Hz, 1 H), 4.08 (d, J = 4.0 Hz, 2 H), 3.84–3.72 (m, 1 H), 2.85–2.61 (m, 4 H), 2.40 (s, 3 H). ¹³C NMR (100 MHz, CDCl₃) δ (ppm): 197.1 (¹³C-enriched), 143.4, 137.4, 137.2, 136.6, 129.8 (2 C), 129.4 (2 C), 128.9 (2 C), 128.8 (2 C), 127.6 (2 C), 127.2 (2 C), 127.0 (2 C), 52.6 (d, J = 1.8 Hz), 46.8 (d, J = 45.2 Hz), 40.8, 33.5, 21.7. HRMS C₂₃H₂₅NO₃S₂ [M+H⁺]; calculated 441.1382, found 441.1398.

N-(4-Hydroxy-1-phenylbutan-2-yl-4⁻¹³C)-4-methylbenzenesulfonamide (Compound 39)

In an argon filled glovebox in a 10 mL flask was added complex 12 (28 mg, 0.05 mmol, 1.0 equiv), NaBH₄ (5.7 mg, 0.15 mmol, 3.0 equiv), THF (1 mL) and MeOH (0.5 mL). The reaction was sealed with a screw cap fitted with a Teflon ® seal, removed from the glovebox and stirred at room temperature for 16 h. The reaction was quenched with 2 M HCl(aq) and extracted with dichloromethane. The combined organic phases were dried over Na₂SO₄, filtered and concentrated in vacuo. Flash column chromatography (25-33% EtOAc in pentane) afforded the product as a pale yellow oil (11 mg, 68%). ¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.69 (d, J = 8.0 Hz, 2 H), 7.26 (d, J = 8.0 Hz, 2 H), 7.21–7.14 (m, 3 H), 6.94 (dd, J = 2.0, 7.8 Hz, 2 H), 4.75 (d, J = 8.4 Hz, 1 H), 4.00 (t, J = 10.8 Hz, 0.5 H), 3.78 (dq, J = 5.3, 11.0 Hz, 0.5 H), 3.66 (ddt, J = 4.0, 9.3, 13.7, 1.5 Hz), 3.42 (dq, J = 5.3, 11.2 Hz, 0.5 H), 2.63 (dq, J = 6.4, 13.6 Hz, 2 H), 2.43 (s, 3 H), 2.22 (bs, 1 H), 1.76 (app. non, 4.4 Hz, 1 H), 1.44 (ddt, 3.6, 10.9, 14.4 Hz, 1 H). ¹³C NMR (100 MHz, CDCl₃) δ (ppm): 143.5, 137.5, 136.9, 129.9 (2 C), 129.4 (2 C), 128.7 (2 C), 127.1 (2 C), 126.8, 63.8 (Unidentified peak), 59.1 (¹³C-enriched), 52.6 (d, J = 1.9 Hz), 41.8 (d, J = 3.1 Hz), 36.8 (d, J = 37.7 Hz), 21.7. HRMS C₁₆¹³CH₂₁NO₃S [M+H⁺]; calculated 321.1348, found 321.1343.
**4-Benzy1-1-tosylazetidin-2-one-2-^{13}C (Compound 40)**

The electrolysis was carried out in a H-Cell separated by a glass frit on an Autolab PGSTAT302 as potentiostat and each chamber was filled with 5 mL 0.2 M TBABF₄ in dichloroethane solution. In the chamber with the working electrode was added complex 12 (28 mg, 0.05 mmol, 1.0 equiv.). The working electrode was a carbon paper electrode (~1 cm²), the reference electrode was a saturated Ag/AgCl, and the counter electrode was a Pt mesh. The electrolysis was carried out at constant potential at 0.3 V vs ferrocene/ferrocene* for 2.2 h. The total charge during the experiment was 3.93 C. After end reaction the reaction mixture was concentrated in vacuo. Flash column chromatography (10-20% EtOAc in pentane) afforded the product as a colorless solid (5 mg, 30%). mp: 136–138 °C.

**1H NMR (400 MHz, CDCl₃) δ (ppm):** 7.93 (d, J = 8.3 Hz, 2 H), 7.38 (d, J = 8.1 Hz, 2 H), 7.34–7.27 (m, 3 H), 7.17 (d, J = 6.7 Hz, 2 H), 4.23 (ddt, J = 9.5, 6.0, 3.2 Hz, 1 H), 3.51 (dd, J = 13.8, 3.6 Hz, 1 H), 2.95–2.80 (m, 2 H), 2.73 (ddd, J = 16.0, 6.6, 3.4 Hz, 1 H), 2.46 (s, 3 H).

**13C NMR (100 MHz, CDCl₃) δ (ppm):** 163.6 ({^{13}C-enriched), 145.5, 136.2, 135.4, 130.2 (2 C), 129.4 (2 C), 129.0 (2 C), 127.6 (2 C), 127.4, 55.1 (d, J = 4.7 Hz), 42.0 (d, J = 42.1 Hz), 39.5 (d, J = 2.0 Hz), 21.9. HRMS C_{16}{^{13}CH_{17}NO_{3}S [M+H]}^{+}; calculated 317.1035, found 317.1043.

**1-Tosyl-2-(2,4,5-trifluorobenzyl)aziridine (Compound 41)**

In a flame-dried round-bottomed flask, 1-bromo-2,4,5-trifluorobenzene (1.1 g, 5.0 mmol, 1.0 equiv) was dissolved in dry THF (5 mL) under an argon atmosphere. The solution was cooled to -10 °C in an ice bath before iPrMgCl·LiCl (1.13 M in THF, 5.1 mmol, 1.02 equiv.) was added dropwise. The reaction was stirred at 0 °C for 1 h. Allyl bromide (449 µL, 5.2 mmol, 1.04 equiv) was then added dropwise and the reaction was stirred for 1 h at 0 °C before reaching room temperature and stirred for additional 2 h. The reaction was quenched with water and acidified to pH = 1 using 2 M HCl(aq). The mixture was extracted with EtOAc and the combined organic phases were washed with brine, dried over MgSO₄, filtered and concentrated under reduced pressure. 1-Allyl-2,4,5-tribromobenzene was isolated as a light yellow oil with minor impurities and was used directly in the following step (606 mg, 70%).

To a round-bottomed flask was added 1-allyl-2,4,5-tribromobenzene (516 mg, 3.0 mmol, 1.0 equiv) in CH₃CN (15 mL). Chloramine-T hydrate (751 mg, 3.3 mmol, 1.1 equiv) and PhNMe₃Br₃ (113 mg, 0.30 mmol, 10 mol%) was added and the
reaction mixture was stirred for 16 h at room temperature. The reaction mixture was concentrated *in vacuo* and the remaining residue was dissolved in CH$_2$Cl$_2$ (10 mL). The solution was filtered through a short silica column eluting with 20% EtOAc in pentane (300 mL). After evaporation of the solvent, the residue was dissolved in CH$_3$CN (10 mL). K$_2$CO$_3$ (2.8 g, 20 mmol, 4.0 equiv) was then added and the mixture was stirred for 2 h at 45 °C. After cooling the reaction to room temperature, the mixture was diluted with Et$_2$O (20 mL). The mixture was filtered through celite and washed with Et$_2$O. Flash column chromatography (5% EtOAc in pentane) afforded the title product as a colorless solid (198 mg, 19%). The spectral data was in accordance with the literature.

mp: 90–92 °C. $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 7.63 (d, $J$ = 8.3 Hz, 2 H), 7.20 (d, $J$ = 8.1 Hz, 2 H), 6.79–6.65 (m, 2 H), 3.05 (dd, $J$ = 14.4, 3.3 Hz, 1 H), 2.89–2.75 (m, 2 H), 2.42 (s, 3 H), 2.27 (dd, $J$ = 8.0 Hz, 1 H), 2.21 (d, $J$ = 4.0 Hz, 1 H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm): 155.6 (ddd, $J$ = 244.5, 9.3, 2.7 Hz), 149.3 (ddd, $J$ = 250.0, 12.3, 2.0 Hz), 147.7 (ddd, $J$ = 250.0, 14.2, 12.3 Hz) 145.0, 134.6, 127.9 (2 C), 127.9 (2 C), 120.8 (dd, $J$ = 18.5, 4.3 Hz), 118.7 (dd, $J$ = 19.1, 5.9 Hz), 105.2 (dd, $J$ = 28.3, 20.8 Hz), 39.9, 32.6, 30.3, 21.5. $^{19}$F NMR (367 MHz, CDCl$_3$) δ (ppm): -119.9 (dd, $J$ = 15.4, 3.4 Hz), -135.5 (dd, $J$ = 21.4, 3.4 Hz), -142.4 (dd, $J$ = 21.5, 15.3 Hz). HRMS C$_{16}$H$_{14}$F$_3$NO$_2$S [M+H$^+$]; calculated 342.0770, found 342.0782.

\[\text{(Phen)Ni\{N(Ts)CH(2,4,5-trifluorobenzyl)CH$_2$\} (Compound 42)}\]

The title compound was prepared according to General Procedure 2 employing 41 (205 mg, 0.60 mmol) yielding the desired product as a dark purple solid (243 mg, 84%). $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 9.90 (d, $J$ = 5.0 Hz, 1 H), 8.47 (d, $J$ = 8.1 Hz, 1 H), 8.40 (d, $J$ = 8.3 Hz, 1 H), 8.25 (d, $J$ = 5.3 Hz, 1 H), 8.20 (d, $J$ = 8.1 Hz, 2 H), 7.96–7.83 (m, 3 H), 7.55 (dd, $J$ = 8.1, 5.2 Hz, 1 H), 7.20 (d, $J$ = 8.0 Hz, 2 H), 7.14 (ddd, $J$ = 11.0, 9.2, 6.8 Hz, 1 H), 6.85–6.76 (m, 1 H), 3.98 (tt, $J$ = 8.9, 4.6 Hz, 1 H), 2.97 (dd, $J$ = 13.5, 4.8 Hz, 1 H), 2.87 (dd, $J$ = 13.4, 8.9 Hz, 1 H), 2.35 (s, 3 H), 0.51 (dd, $J$ = 8.3, 5.5 Hz, 1 H), 0.25 (app. t, $J$ = 5.1 Hz, 1 H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm): 156.8 (d, $J$ = 243.8, 7.6 Hz), 153.1, 149.4, 148.5 (d, $J$ = 247.2 Hz), 147.2, 146.8 (ddd, $J$ = 240.2, 13.3, 3.3 Hz), 144.3, 143.0, 141.0, 137.1, 136.3, 129.3, 129.3 (2 C), 128.7, 127.7 (2 C), 127.5, 126.5, 126.4, 125.4, 123.9 (d, $J$ = 19.3 Hz), 119.8 (dd, $J$ = 18.5, 6.5 Hz), 105.1 (dd, $J$ =29.2, 20.6 Hz), 65.5, 38.4, 21.6, -6.9. $^{19}$F NMR (367
MHz, CD$_2$Cl$_2$) $\delta$ (ppm): -119.4 (dd, $J$ = 15.2, 2.7 Hz), -139.1 (dd, $J$ = 21.6, 2.7 Hz), -145.4 (dd, $J$ = 21.7, 15.4 Hz).

(Phen)Ni{N(Ts)CH(2,4,5-trifluorobenzyl)CH$_2^{13}$CO} (Compound 43)

The title compound was prepared according to General Procedure 3 employing 42 (145 mg, 0.5 mmol) yielding the desired product as a bright red solid (268 mg, 89%) with minor dichloroethane impurities.

$^1$H NMR (400 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 9.59 (d, $J$ = 4.3 Hz, 1 H), 9.27 (d, $J$ = 5.0 Hz, 2 H), 8.49 (d, $J$ = 8.1 Hz, 1 H), 8.09–7.90 (m, 4 H), 7.86 (dd, $J$ = 8.1, 5.2 Hz, 2 H), 7.21 (d, $J$ = 7.9 Hz, 2 H), 7.11 (ddd, $J$ = 13.8, 8.5 Hz, 1 H), 3.36 (dd, $J$ = 13.8, 8.5 Hz, 1 H), 3.15–3.04 (m, 1 H), 2.95 (dd, $J$ = 13.8, 5.3 Hz, 1 H), 2.40 (s, 3 H), 2.25 (ddd, $J$ = 16.2, 4.9, 1.1 Hz, 1 H), 2.15 (app. dt, $J$ = 16.0, 6.8 Hz, 1 H).

$^{13}$C NMR (126 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): 239.2 ($^{13}$C-enriched), 156.6 (ddd, $J$ = 244.0, 9.5, 2.4 Hz), 152.7, 151.4, 148.7 (ddd, $J$ = 251.6, 18.4, 11.0 Hz), 147.0, 146.6 (ddd, $J$ = 242.9, 18.4, 3.5 Hz), 144.4, 142.0, 141.5, 138.0, 137.8, 129.2 (2 C), 129.1, 128.4, 127.4 (2 C), 126.8, 126.6, 125.1, 124.9, 123.5 (ddd, $J$ = 18.4, 5.7, 4.1 Hz), 120.2 (dd, $J$ = 18.7, 6.4 Hz), 105.1 (dd, $J$ = 29.0, 20.6 Hz), 60.1 (d, $J$ = 20.0 Hz), 38.4, 30.1, 21.5. $^{19}$F NMR (376 MHz, CD$_2$Cl$_2$) $\delta$ (ppm): -118.9 (dd, $J$ = 15.2, 3.1 Hz), -138.3 (dd, $J$ = 21.6, 3.1 Hz), -145.2 (dd, $J$ = 21.6, 15.3 Hz).

4-Methyl-N-(4-oxo-4-(3-(trifluoromethyl)-5,6-dihydro-[1,2,4]triazolo[4,3-a]pyrazin-7(8H)-yl)-1-(2,4,5-trifluorophenyl)butan-2-yl-13$^{13}$C)benzenesulfonamide (Compound 46)

43 (122 mg, 0.20 mmol, 1.0 equiv) was dissolved in dichloroethane (5 mL) in a vial. Under air was added 4 M HCl$_{(aq)}$ (2 mL) and the reaction was stirred at room temperature for 1 h. Formic acid (2 mL) was added and the reaction stirred for additional 3 h. The reaction was extracted with dichloromethane and the combined organic phases were dried over Na$_2$SO$_4$, filtered and concentrated in vacuo. The remaining residue was dissolved in dichloromethane (5 mL) and dropwise added to a solution of EDC·HCl (77 mg, 0.40 mmol, 2.0 equiv), DMAP (98 mg, 0.80 mmol, 4.0 equiv) and 3-(trifluoromethyl)-5,6,7,8-tetrahydro-[1,2,4]triazolo[4,3-a]pyrazine hydrochloride (45) (50 mg, 0.22 mmol, 1.1 equiv) in dichloromethane (5 mL). The reaction was stirred at room temperature
for 16 h. The reaction was quenched with 4 M HCl\(_{(aq)}\) and acidified to pH = 1 before being extracted with dichloromethane. The combined organic phases were dried over Na\(_2\)SO\(_4\), filtered and concentrated \textit{in vacuo}. Flash column chromatography (2 → 5% MeOH in CH\(_2\)Cl\(_2\)) afforded the product as a colorless solid (96 mg, 85%). The product was isolated as a mixture of rotamers (1:1). mp: 210–214 °C. \(^1\)H NMR (400 MHz, CD\(_3\)CN) \(\delta\) (ppm): 7.44 (app t, \(J = 7.1\) Hz, 4 H), 7.18 (d, \(J = 8.0\) Hz, 4 H), 7.03–6.90 (m, 2 H), 6.89–6.76 (m, 2 H), 6.00–5.87 (m, 2 H), 4.93–4.74 (m, 4 H), 4.19–4.05 (m, 4 H), 4.04–3.70 (m, 6 H), 2.85–2.59 (m, 8 H), 2.35 (s, 6 H). \(^13\)C NMR (100 MHz, DMSO-\(d_6\)) \(\delta\) (ppm): 168.9 (13C-enriched), 168.8 (13C-enriched), 155.6 (ddd, \(J = 3.3, 11.8\) Hz, 246.3 2 C), 150.9, 150.7, 147.9 (m, 2 C), 145.2 (m, 2 C), 142.6 (2 C), 142.2 (2 C), 138.3, 138.2, 129.1 (4 C), 125.9 (4 C), 121.8 (m, 2 C), 119.2 (m, 2 C), 118.4 (q, \(J = 268.3\) Hz, 2 C), 105.3 (ddd, \(J = 2.2, 23.0, 31.0\) Hz, 2 C), 51.2 (2 C), 43.5, 42.9, 42.0, 41.1, 38.6, 38.3 (2 C), 37.4, 33.3, 31.1, 20.8 (2 C). \(^19\)F NMR (367 MHz, CD\(_3\)CN) \(\delta\) (ppm) -63.7, -63.8, -120.2 (dd, \(J = 15.3, 3.5\) Hz), -120.4 (dd, \(J = 15.2, 3.4\) Hz), -138.2 (dd, \(J = 12.3, 3.5\) Hz), 138.3 (dd, \(J = 12.3, 3.5\) Hz), -145.2 (dd, \(J = 21.2, 9.0\) Hz), -145.3 (dd, \(J = 21.2, 9.0\) Hz). HRMS C\(_{22}\)\(^{13}\)CH\(_2\)F\(_6\)N\(_5\)O\(_3\)S [M+H\(^+\)]; calculated 563.1376, found 563.1390.

**References**


Spectra (\(^1\)H NMR and \(^{13}\)C NMR) of 1a and 1a’

1a and 1a’
\(^1\)H NMR, 400 MHz, CDCl\(_3\)

1a and 1a’
\(^{13}\)C NMR, 100 MHz, CDCl\(_3\)

Spectra (\(^1\)H NMR and \(^{13}\)C NMR) of 1a and 1a’
Spectra ($^1$H NMR and $^{13}$C NMR) of 2a

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

(1$^{13}$C NMR, 100 MHz, CDCl$_3$)

Spectra ($^1$H NMR and $^{13}$C NMR) of 2a
Spectra ($^1$H NMR and $^{13}$C NMR) of 3a

3a

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

3a

($^{13}$C NMR, 100 MHz, CDCl$_3$)

Spectra ($^1$H NMR and $^{13}$C NMR) of 3a
Spectra (\(^1\)H NMR and \(^{13}\)C NMR) of 4a

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

\(^{13}\)C NMR, 100 MHz, CDCl\(_3\)

Spectra (\(^1\)H NMR and \(^{13}\)C NMR) of 4a
Spectra (\(^1\)H NMR and \(^{13}\)C NMR) of 5a

\((\text{\(^1\)H NMR, 400 MHz, CDCl}_3)\)

\((\text{\(^{13}\)C NMR, 100 MHz, CDCl}_3)\)

Spectra (\(^1\)H NMR and \(^{13}\)C NMR) of 5a
Spectra (\(^1\)H NMR and \(^{13}\)C NMR) of 6a

\[ 6a \quad (\text{\(^1\)H NMR, 400 MHz, CDCl}_3) \]

\[ 6a \quad (\text{\(^{13}\)C NMR, 100 MHz, CDCl}_3) \]

Spectra (\(^1\)H NMR and \(^{13}\)C NMR) of 6a
Spectra (1H NMR and 13C NMR) of 7a.

1H NMR, 400 MHz, CDCl₃

13C NMR, 100 MHz, CDCl₃
Spectra (\(^1\)H NMR and \(^{13}\)C NMR) of 8a

\(\text{\textbf{8a}}\)

\((\text{\textbf{1}H NMR, 400 MHz, C}_3\text{D}_2\text{O})\)

\(\text{\textbf{8a}}\)

\((\text{\textbf{13}C NMR, 100 MHz, C}_3\text{D}_2\text{O})\)

Spectra (\(^1\)H NMR and \(^{13}\)C NMR) of 8a
Spectra (\(^1\)H NMR and \(^{13}\)C NMR) of 9a

Spectra (\(^1\)H NMR and \(^{13}\)C NMR) of 9a
$^{1}H$ NMR, 400 MHz, CDCl$_3$`

$^{13}C$ NMR, 100 MHz, CDCl$_3$`
Spectra (\(^1\)H NMR, \(^{13}\)C NMR and \(^{19}\)F NMR) of 10a

\[ 10a \]

\[ (^{19}\text{F NMR}, 367 \text{ MHz, CDCl}_3) \]
(1H NMR, 400 MHz, CDCl₃)

(13C NMR, 100 MHz, CDCl₃)
Spectra (\(^1\)H NMR, \(^{13}\)C NMR and \(^{19}\)F NMR) of 11a

\[ \text{11a} \]

\((^{19}\text{F NMR, 367 MHz, CDCl}_3)\)
Spectra (\(^1\)H NMR and \(^{13}\)C NMR) of 1 and 1'.

\(1\) and 1' (\(^1\)H NMR, 400 MHz, \(\text{CD}_2\text{Cl}_2\))

\(1\) (\(^{13}\)C NMR, 100 MHz, \(\text{CD}_2\text{Cl}_2\))

Spectra (\(^1\)H NMR and \(^{13}\)C NMR) of 1 and 1'
Spectra ($^1$H NMR and $^{13}$C NMR) of 2

$\left( ^{1}H \text{ NMR, } 400 \text{ MHz, } \text{CD}_2\text{Cl}_2 \right)$

$\left( ^{13}C \text{ NMR, } 100 \text{ MHz, } \text{CD}_2\text{Cl}_2 \right)$

Spectra ($^1$H NMR and $^{13}$C NMR) of 2
Spectra ($^1$H NMR and $^{13}$C NMR) of 3
Spectra (\(^1\)H NMR and \(^{13}\)C NMR) of 4
Spectra (\(^1\)H NMR and \(^{13}\)C NMR) of 5

(\(^1\)H NMR, 400 MHz, \(\text{CD}_2\text{Cl}_2\))

(\(^{13}\)C NMR, 100 MHz, \(\text{CD}_2\text{Cl}_2\))

Spectra (\(^1\)H NMR and \(^{13}\)C NMR) of 5
Spectra ($^1$H NMR and $^{13}$C NMR) of 6
Spectra (1H NMR and 13C NMR) of 7

(1H NMR, 400 MHz, CD2Cl2)

(13C NMR, 100 MHz, CD2Cl2)
Spectra ($^1$H NMR and $^{13}$C NMR) of 8
Spectra ($^1$H NMR and $^{13}$C NMR) of 9
(1H NMR, 400 MHz, CD$_2$Cl$_2$)

(13C NMR, 100 MHz, CD$_2$Cl$_2$)
Spectra (\(^1\)H NMR, \(^{13}\)C NMR and \(^{19}\)F NMR) of 10

\((^{19}\)F NMR, 367 MHz, CD\(_2\)Cl\(_2\))
S62

(1H NMR, 400 MHz, CD$_2$Cl$_2$)

11

(F$_3$C=S=O, Ni-Ni)

(13C NMR, 100 MHz, CD$_2$Cl$_2$)

11

(F$_3$C=S=O, Ni-Ni)
Spectra ($^1$H NMR, $^{13}$C NMR and $^{19}$F NMR) of 11

Spectra ($^1$H NMR, $^{13}$C NMR and $^{19}$F NMR) of 11
12 and 12' (\(^1\)H NMR, 400 MHz, CD\(_2\)Cl\(_2\))

After 14 days in air
Spectra ($^1$H NMR and $^{13}$C NMR) of 12 and 12' ($^{13}$C NMR, 126 MHz, CD$_2$Cl$_2$)
Spectra ($^1$H NMR and $^{13}$C NMR) of 13
Spectra (\(^1\text{H NMR}\) and \(^{13}\text{C NMR}\)) of 14 

\begin{align*}
\text{14} \quad \text{N} \quad \text{Ni} \quad \text{N} \quad \text{Ts} \\
\text{O} \quad \text{N} \quad \text{Ni} \quad \text{N} \quad \text{Ts} \\
\text{14} \quad \text{N} \quad \text{Ni} \quad \text{N} \quad \text{Ts} \\
\text{O} \quad \text{N} \quad \text{Ni} \quad \text{N} \quad \text{Ts}
\end{align*}

\(^1\text{H NMR, 400 MHz, CD}_2\text{Cl}_2\)

\(^{13}\text{C NMR, 126 MHz, CD}_2\text{Cl}_2\)
Spectra (\(^1\)H NMR and \(^{13}\)C NMR) of 15
Spectra (\(^1\)H NMR and \(^{13}\)C NMR) of 16

16

(\(^1\)H NMR, 400 MHz, CD\(_2\)Cl\(_2\))

\(\text{13C NMR, 126 MHz, CD}_2\text{Cl}_2\)

Spectra (\(^1\)H NMR and \(^{13}\)C NMR) of 16
Spectra ($^1$H NMR and $^{13}$C NMR) of 17

($^1$H NMR, 400 MHz, CD$_2$Cl$_2$)

($^{13}$C NMR, 126 MHz, CD$_2$Cl$_2$)
Spectra ($^1$H NMR and $^{13}$C NMR) of 18

(1H NMR, 400 MHz, CD$_2$Cl$_2$)

(13C NMR, 126 MHz, CD$_2$Cl$_2$)
Spectra ($^1$H NMR and $^{13}$C NMR) of 19

$^{13}$C NMR, 126 MHz, CD$_2$Cl$_2$
\[ \text{S73} \]

\((^1\text{H NMR, 400 MHz, CD}_2\text{Cl}_2)\)

\[ \text{20} \]

\((^{13}\text{C NMR, 126 MHz, CD}_2\text{Cl}_2)\)
Spectra (\(^1\)H NMR, \(^{13}\)C NMR and \(^{19}\)F NMR) of 20

\[
\text{(}^{19}\text{F NMR, 367 MHz, CD}_2\text{Cl}_2)\]

Spectra (\(^1\)H NMR, \(^{13}\)C NMR and \(^{19}\)F NMR) of 20
Spectra ($^1$H NMR, $^{13}$C NMR and $^{19}$F NMR) of 21

$^{19}$F NMR, 367 MHz, CD$_2$Cl$_2$
Spectra ($^1$H NMR and $^{13}$C NMR) of 22
Spectra (\(^1\)H NMR and \(^{13}\)C NMR) of 23 and 23’

\[
\begin{align*}
\text{23 and 23’} \\
\text{(\(^1\)H NMR, 400 MHz, CDCl}_3) \\
\end{align*}
\]

\[
\begin{align*}
\text{23 and 23’} \\
\text{\(^{13}\)C NMR, 100 MHz, CDCl}_3)
\end{align*}
\]
Spectra ($^1$H NMR and $^{13}$C NMR) of 24
Spectra ($^1$H NMR and $^{13}$C NMR) of 25
Spectra ($^1$H NMR and $^{13}$C NMR) of 26

26

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

26

($^{13}$C NMR, 100 MHz, CDCl$_3$)
Spectra ($^1$H NMR and $^{13}$C NMR) of 27
Spectra ($^1$H NMR and $^{13}$C NMR) of 28
Spectra (\textsuperscript{1}H NMR and \textsuperscript{13}C NMR) of 29
(\(^{1}H\) NMR, 400 MHz, CDCl\(_3\))

(\(^{13}C\) NMR, 100 MHz, CDCl\(_3\))
Spectra ($^1$H NMR, $^{13}$C NMR and $^{19}$F NMR) of 30
Spectra ($^1$H NMR and $^{13}$C NMR) of 31
Spectra (\(^1\)H NMR and \(^{13}\)C NMR) of 32
S33

$^{1}H$ NMR, 400 MHz, CDCl$_3$

33

$^{13}C$ NMR, 100 MHz, CDCl$_3$
Spectra ($^1$H NMR, $^{13}$C NMR and $^{19}$F NMR) of 33

(19F NMR, 367 MHz, CDCl$_3$)
Spectra (\(^1\)H NMR and \(^{13}\)C NMR) of 34
Spectra ($^1$H NMR and $^{13}$C NMR) of 35
Spectra (\(^1\)H NMR and \(^{13}\)C NMR) of 36
Spectra ($^1$H NMR and $^{13}$C NMR) of 37
Spectra ($^1$H NMR and $^{13}$C NMR) of 38
Spectra ($^1$H NMR and $^{13}$C NMR) of 39
Spectra ($^1$H NMR and $^{13}$C NMR) of 40
(\textsuperscript{1}H NMR, 400 MHz, CDCl\textsubscript{3})

\textbf{41}

\textsuperscript{13}C NMR, 100 MHz, CDCl\textsubscript{3})

\textbf{41}
Spectra of \(41\):

- **\(^{1}H\) NMR**: 367 MHz, CDCl\(_3\)
- **\(^{19}F\) NMR**: 367 MHz, CDCl\(_3\)
- **\(^{13}C\) NMR**: 367 MHz, CDCl\(_3\)

Diagram of **41**

Spectra (\(^{1}H\) NMR, \(^{13}C\) NMR and \(^{19}F\) NMR) of **41**
42

(\(^1\)H NMR, 400 MHz, CD\(_2\)Cl\(_2\))

42

(\(^{13}\)C NMR, 126 MHz, CD\(_2\)Cl\(_2\))
Spectra (\textsuperscript{1}H NMR, \textsuperscript{13}C NMR and \textsuperscript{19}F NMR) of 42

\textsuperscript{19}F NMR, 367 MHz, CD\textsubscript{2}Cl\textsubscript{2}
(1H NMR, 400 MHz, CD₂Cl₂)

(13C NMR, 126 MHz, CD₂Cl₂)
Spectra ($^1$H NMR, $^{13}$C NMR and $^{19}$F NMR) of 43
(1H NMR, 400 MHz, CD$_3$CN)

(13C NMR, 100 MHz, DMSO-$d_6$)
Spectra (\textsuperscript{1}H NMR, \textsuperscript{13}C NMR and \textsuperscript{19}F NMR) of 46

(\textsuperscript{19}F NMR, 367 MHz, CD\textsubscript{3}CN)

Spectra (\textsuperscript{1}H NMR, \textsuperscript{13}C NMR and \textsuperscript{19}F NMR) of 46