Hydrophobically Assisted Separation-Friendly Mitsunobu Reaction

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I. General information

Unless otherwise noted, all reactions were carried out in oven-dried glassware under an atmosphere of argon. Tetrahydrofuran was dried and distilled from sodium-potassium alloy. Dichloromethane was distilled from calcium hydride. Methanol was dried under reflux with magnesium and then distilled. \(N, N\)-Dimethylformamide was dried over calcium hydride and distilled under vacuum. Reactions were monitored by analytical thin-layer chromatography (TLC) on Merck silica gel 60 F\textsubscript{254} plates (0.25 mm), visualized by ultraviolet light and/ or by staining with ceric ammonium molybdate. \(^1\)H NMR spectra were obtained on Bruker AVANCE III 400 spectrometer at ambient temperature. Data were reported as follows: chemical shift on the \(\delta\) scale (using either TMS or residual proton solvent as internal standard), multiplicity (br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), integration, and coupling constant(s) in hertz. \(^{13}\)C NMR spectra were obtained with proton decoupling on a Bruker AVANCE III 400 (100 MHz) spectrometer and were reported in ppm with residual solvent for internal standard. High resolution mass spectra were obtained on a Thermo Scientific LTQ Orbitrap Discovery spectrometer or Waters Xevo G2 Q-TOF mass spectrometer. Mass spectra were obtained on a Bruker Ultraflex MALDI TOF spectrometer or Finnigan TRACE DSQ spectrometer. Elemental analysis data were recorded on a PE-2400C elemental analyzer. Melting point was determined by WRS-2A Digital Melting Point Apparatus.
II. Synthesis of hydrophobic-tag acids

Synthesis of methyl 4-(octadecylthio)benzoate (S1):

Cesium carbonate (2.16 g, 6.63 mmol) was placed in a 100 mL-reaction flask under argon, DMSO (20 mL), methyl 4-nitrobenzoate (1.00 g, 5.52 mmol) and octadecane-1-thiol (1.90 g, 6.63 mmol) was added at r.t. under argon. Then the reaction flask was sealed up with rubber plug and irradiated under ultrasonic waves in ultrasonic cleaner. After 5 min, water (50 mL) was added, and the product was extracted with hexane–ethyl acetate (10/1, 60 mL×3). The combined organic layer was dried over sodium sulfate. Concentration followed by purification on silica gel (petroleum ether/ethyl acetate = 15/1) afforded methyl 4-(octadecylthio)benzoate (S1, 2.10 g, 90%) as a white solid, m.p. 81-82 °C. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.92 (d, $J = 8.5$ Hz, 2H), 7.27 (d, $J = 8.4$ Hz, 2H), 3.89 (s, 3H), 2.97 (t, $J = 7.4$ Hz, 2H), 1.72 – 1.65 (m, 2H), 1.47 – 1.40 (m, 2H), 1.25 (brs, 28H), 0.88 (t, $J = 6.7$ Hz, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 166.85, 144.56, 129.89, 126.49, 126.24, 52.01, 31.94, 29.71, 29.68, 29.65, 29.58, 29.49, 29.38, 29.16, 28.91, 28.75, 22.71, 14.13. MS (EI) Calcd. for C$_{26}$H$_{44}$O$_2$S [M]$^+$ 420.3, found 420.3. Elemental Analysis: Calcd. for C$_{26}$H$_{44}$O$_2$S C 74.23%, H 10.54%; Found C 74.15%, H 10.46%.

Synthesis of methyl 4-(octadecylsulfonyl)benzoate (S2):

To a stirred solution of methyl 4-(octadecylthio)benzoate (1.00 g, 2.38 mmol) in dichloromethane (30 mL) was added 70% mCPBA (1.30 g, 5.24 mmol) at r.t. After 5 min, saturated sodium metabisulfite was added to quench the reaction and stirred for 15 min. Then the mixture was extract with dichloromethane (50 mL×3), the organic layer was washed with saturated sodium bicarbonate three times, brine, and concentrated to afford white solids, which are pure enough for characterization and the next step reaction (1.10 g, 100%), m.p. 91-92 °C. $^1$H NMR (400 MHz, CDCl$_3$) δ 8.24 (d, $J = 8.5$ Hz, 2H), 8.00 (d, $J = 8.4$ Hz, 2H), 3.98 (s, 3H), 3.10 (t, $J = 7.4$ Hz, 2H), 1.74 - 1.68 (m, 2H), 1.36 – 1.22 (m, 29H), 0.88 (t, $J = 6.7$ Hz, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 165.51, 143.02, 134.78, 130.41, 128.20, 56.20, 52.74, 31.93, 29.69, 29.66, 29.63, 29.55, 29.45,
29.36, 29.21, 28.97, 28.24, 22.69, 22.57, 14.13. MS (ESI) Calcd. for C_{26}H_{45}O_{4}S [M+H]^+ 453.3, found 453.4. Elemental Analysis: Calcd. for C_{26}H_{44}O_{4}S C 68.98%, H 9.80%; Found C 68.89%, H 9.79%.

**Synthesis of 4-(octadecylsulfonyl)benzoic acid (7):**

$$\text{C}_{18}\text{H}_{37}\text{SO}$$

To a solution of methyl 4-(octadecylsulfonyl)benzoate (1.00 g, 2.21 mmol) in THF/H_2O (20 mL, v/v = 7/1) was added lithium hydroxide monohydrate (0.37 g, 8.84 mmol), stirred overnight, and the reaction mixture was acidified to below pH 2 using 1 M HCl. The white solids were filtrated, washed with water and dried in vacuo to afford the acid 7 (0.93 g, 96%), m.p. 162-164 °C. ¹H NMR (400 MHz, Pyridine-d5) δ 10.44 (brs, 1H), 8.56 (d, J = 8.4 Hz, 2H), 8.32 (d, J = 8.4 Hz, 2H), 3.45 (t, J = 8.0 Hz, 2H), 1.87 – 1.80 (m, 2H), 1.33 – 1.18 (m, 30H), 0.86 (t, J = 6.8 Hz, 3H) ¹³C NMR (100 MHz, Pyridine-d5) δ 167.58, 143.27, 137.23, 130.71, 128.37, 55.79, 31.88, 29.74, 29.70, 29.68, 29.63, 29.54, 29.37, 29.30, 29.00, 28.21, 22.78, 22.70, 14.04. MS (EI) Calcd. for C_{25}H_{42}O_{4}S [M-H] - 437.3, found 437.3. Elemental Analysis: Calcd. for C_{25}H_{42}O_{4} C 68.45%, H 9.65%; Found C 68.23%, 9.71%.

**Synthesis of pentatriacontan-18-ol (S3):**

$$\text{C}_{16}\text{H}_{33}\text{O}$$

Pentatriacontan-18-one (2.00 g, 3.94 mmol) was dissolved in THF (120 mL) at 40 °C. Lithium aluminum hydride (300 mg, 7.89 mmol) was added in portions. The reaction was stirred for 30 min at 40 °C. Solid sodium sulfate decahydrate was added and the slurry was stirred for 30 min. The solids were filtered off, and the filtrate was diluted with 1 M HCl, extracted with heptane/ethyl acetate (v/v = 10/1, 80 mL×3). The organic phase was dried over sodium sulfate and concentrated to give white solids. The crude product is pure enough without further purification (1.80 g, 90%). ¹H NMR (400 MHz, CDCl₃) δ 3.58 (s, 1H), 1.42 – 1.20 (m, 64H), 0.88 (t, J = 6.5 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 72.03, 37.48, 31.92, 29.69, 29.36, 25.65, 22.69, 14.12. The data are identical with those reported in the literature.¹

**Synthesis of pentatriacontan-18-yl methanesulfonate (10):**
To a solution of pentatriacontan-18-ol (1.00 g, 1.96 mmol) and triethylamine (0.71 mL, 5.11 mmol) in DCM/THF (80 mL, v/v = 1/1) at r.t., was added methanesulfonyl chloride (0.20 mL, 2.57 mmol). After the mixture was stirred at 35 °C for 2 hours, the reaction mixture was concentrated to 1/2 of its original volume, diluted with heptane (100 mL), washed successively with 1 M HCl, saturated sodium bicarbonate and brine, dried over sodium sulfate, and concentrated to yellow wax. The crude product is pure enough without further purification for characterization and next step reaction (1.10 g, 98%). H NMR (400 MHz, CDCl₃) δ 4.80 – 4.60 (m, 1H), 2.99 (s, 3H), 1.68 (s, 4H), 1.44 – 1.15 (m, 60H), 0.88 (t, J = 6.3 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 84.42, 38.69, 34.46, 31.93, 29.70, 29.57, 29.47, 29.41, 29.37, 24.97, 22.70, 14.13. HRMS: Calcd. for C₃₆H₇₈NO₃S [M+NH₄]+ 604.5702, found 604.5695.

**Synthesis of methyl 4-(pentatriacontan-18-ylthio)benzoate (11):**

Cesium carbonate (1.10 g, 3.41 mmol), pentatriacontan-18-yl methanesulfonate (1.00 g, 1.70 mmol), methyl 4-mercaptobenzoate² (0.46 g, 3.41 mmol) were added into DMF (10 mL) under argon. The mixture was stirred at 80 °C overnight. After the solution cooled to r.t., diluted with heptane/ethyl acetate (v/v = 10/1, 80 mL), washed with water, 1 M HCl, brine, dried over sodium sulfate, and concentrated to afford a yellow oil, which was purified by column chromatography (heptane/ethyl acetate = 50/1) to give an colorless oil (1.00 g, 95%). H NMR (400 MHz, CDCl₃) δ 7.92 (d, J = 8.3 Hz, 2H), 7.33 (d, J = 8.3 Hz, 2H), 3.89 (s, 3H), 3.27 – 3.21 (m, 1H), 1.65 -1.57 (m, 4H), 1.43 – 1.25 (m, 60H), 0.88 (t, J = 6.5 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 166.78, 144.00, 129.82, 128.56, 127.06, 51.95, 47.79, 34.50, 31.93, 29.70, 29.67, 29.58, 29.53, 29.51, 29.36, 26.76, 22.68, 14.09. HRMS: Calcd for C₄₃H₇₉O₂S [M+H]+ 659.5801, found 659.5797.

**Synthesis of methyl 4-(pentatriacontan-18-ylsulfonyl)benzoate (12):**

To a stirred solution of methyl 4-(pentatriacontan-18-ylthio)benzoate (1.00 g, 1.52 mmol) in dichloromethane (30 mL) was added 70% mCPBA (0.82 g, 3.34 mmol) at r.t. After 5 min,
saturated sodium metabisulfite was added to quench the reaction and stirred for 15 min. Then the mixture was extract with dichloromethane (50 mL×3). The organic layer was washed with saturated sodium bicarbonate three times, brine, and concentrated to white solids, which are pure enough for characterization and the next step reaction (1.00 g, 100%), m.p. 61-62 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.21 (d, J = 8.0 Hz, 2H), 7.96 (d, J = 8.1 Hz, 2H), 3.97 (s, 3H), 2.92 (br.s, 1H), 1.89 – 1.71 (m, 2H), 1.59 – 1.52 (m, 2H), 1.42-1.22 (m, 60H), 0.88 (t, J = 6.0 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 165.56, 142.30, 134.61, 130.17, 128.90, 64.60, 52.69, 31.93, 29.70, 29.59, 29.49, 29.46, 29.36, 29.23, 27.79, 26.70, 22.69, 14.11. HRMS: Calcd for C₄₃H₇₉O₄S [M+H]⁺ 691.5699, found 691.5690.

**Synthesis of 4-(pentatriacontan-18-ylsulfonyl)benzoic acid (13):**

![Chemical Structure Image]

To a solution of methyl 4-(pentatriacontan-18-ylsulfonyl)benzoate (1.00 g, 1.45 mmol) in THF/H₂O (20 mL, v/v = 7/1) was added lithium hydroxide monohydrate (0.24 g, 5.78 mmol), the mixture was stirred overnight. Then the reaction mixture was acidified to below pH 2 using 1 M HCl, cooled to 0 °C and stayed until white solids appeared. The white solids were filtrated, washed with water and dried in vacuum to afford the acid 13 (0.93 g, 95%), m.p. 82-83 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.31 (d, J = 8.0 Hz, 2H), 8.02 (d, J = 8.1 Hz, 2H), 2.94 (s, 1H), 1.91 – 1.70 (m, 2H), 1.67 – 1.52 (m, 2H), 1.50 – 1.10 (m, 60H), 0.88 (t, J = 6.0 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 170.04, 143.25, 133.58, 130.80, 129.07, 64.63, 31.93, 29.70, 29.60, 29.50, 29.46, 29.37, 29.23, 27.78, 26.71, 22.69, 14.12. HRMS: Calcd. for C₄₂H₇₅O₄S [M-H]⁻ 676.5386, found 675.5355.

**III. Mitsunobu reaction and characterization of products**

**General procedure for Mitsunobu reaction and purification by C-18 SPE (take the synthesis of 14h as an example):**

To a solution of the acid 13 (68.0 mg, 0.1 mmol), the alcohol h (67.0 mg, 0.12 mmol), and triphenylphosphine (53.0 mg, 0.2 mmol) in THF (6 mL) under argon was added DEAD (37.0 mg,
0.21 mmol) dropwise. The mixture was stirred for 1 hour at 0°C and another 2 hours at room temperature. Then C-18 silica gel (500 mg) was added to the reaction vessel and the solvents were removed. The dry C-18 silica gel with the adsorbed reaction mixture on it was transferred to a 1.8 cm diameter column, which was filled with 1 cm high C-18 silica gel in advance. The C-18 silica gel was washed with methanol/water (2:1, 30 mL), methanol (20 mL×2), and acetone (30 mL) under reduced pressure. The filtrates containing the desorbed product were collected and concentrated to give colorless oil **14h** (114 mg, 94% yield).

**General procedure for Mitsunobu reaction and purification by silica gel filtration (take the synthesis of 8g as an example):**

To a solution of the acid **7** (77.0 mg, 0.17 mmol), the alcohol **g** (21.0 mg, 0.13 mmol), and triphenylphosphine (67.0 mg, 0.26 mmol) in THF (6 mL) under argon was added DEAD (47.0 mg, 0.27 mmol) dropwise. The mixture was stirred at 0°C for 1 hour and another 2 hours at room temperature. Then silica gel (1.00 g) was added to the reaction vessel and the solvents were removed. The silica gel with the adsorbed reaction mixture on it was transferred to a 2.5 cm diameter column which was filled with 5 cm high silica gel in advance, and washed with ethyl acetate/petroleum ether (v/v = 1/8). The filtrates containing the product were collected and concentrated to give **8g** as white solids (67.0 mg, 88%).

**Ethyl 4-(dodecyloxy)benzoate (2a):**

![Ethyl 4-(dodecyloxy)benzoate (2a)](image)

Yield: 95%, white solids. ¹H NMR (400 MHz, CDCl₃) δ 7.99 (d, J = 8.8 Hz, 2H), 6.90 (d, J = 8.8 Hz, 2H), 4.35 (q, J = 7.1 Hz, 2H), 3.99 (t, J = 6.5 Hz, 2H), 1.82 – 1.75 (m, 2H), 1.47 – 1.26 (m, 21H), 0.88 (t, J = 6.8 Hz, 3H). The data are identical with those reported in the literature.³

**Tetrahydrofuran-3-yl 4-(dodecyloxy)benzoate (2b):**

![Tetrahydrofuran-3-yl 4-(dodecyloxy)benzoate (2b)](image)

Yield: 92%, yellow wax. ¹H NMR (400 MHz, CDCl₃) δ 7.99 (d, J = 8.8 Hz, 2H), 6.91 (d, J = 9.2 Hz, 2H), 5.53 – 5.50 (m, 1H), 4.03 – 3.90 (m, 6H), 2.31 - 2.22 (m, 1H), 2.17 –2.11 (m, 1H), 1.83 – 1.76 (m, 2H), 1.48 – 1.42 (m, 2H), 1.40 – 1.21 (m, 16H), 0.88 (t, J = 6.8 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 166.05, 163.11, 131.60, 122.09, 114.04, 74.95, 73.23, 68.20, 67.11, 32.95, 31.87,
29.61, 29.59, 29.54, 29.51, 29.31, 29.06, 25.93, 22.65, 14.07. HRMS: Calcd. for C_{23}H_{37}O_{4} [M+H]^+ 377.2692, found 377.2685. Elemental Analysis: Calcd. for C_{23}H_{36}O_{4} C, 73.37; H, 9.64; found C, 73.16, H, 9.59.

3-Butyn-2-yl 4-(dodecyloxy)benzoate (2c):

\[
\text{C}_{12}\text{H}_{25}\text{O} = \text{O} = \text{O}
\]

Yield: 90%, white wax. $^1$H NMR (400 MHz, CDCl$_3$) δ 8.02 (d, $J = 8.8$ Hz, 2H), 6.91 (d, $J = 8.8$ Hz, 2H), 5.68 (dq, $J = 6.7$, 3.3 Hz, 1H), 4.00 (t, $J = 6.5$ Hz, 2H), 2.47 (d, $J = 1.9$ Hz, 1H), 1.83 – 1.76 (m, 2H), 1.63 (d, $J = 8.7$ Hz, 3H), 1.47 – 1.44 (m, 2H), 1.32 - 1.26 (m, 17H), 0.88 (t, $J = 6.7$ Hz, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 165.19, 163.19, 131.60, 121.76, 114.07, 82.43, 72.77, 68.22, 60.18, 31.90, 29.63, 29.61, 29.56, 29.53, 29.33, 29.08, 25.95, 22.67, 21.36, 14.10. HRMS: Calcd. for C$_{23}$H$_{35}$O$_3$ [M+H]$^+$ 359.2586, found 359.2615.

1-Phenylethyl 4-(dodecyloxy)benzoate (2d):

\[
\text{C}_{12}\text{H}_{25}\text{O} = \text{O} = \text{O}
\]

Yield: 91%, white wax. $^1$H NMR (400 MHz, CDCl$_3$) δ 8.02 (d, $J = 8.8$ Hz, 2H), 7.44 (d, $J = 7.3$ Hz, 2H), 7.36 (t, $J = 7.4$ Hz, 2H), 7.28 (t, $J = 7.2$ Hz, 1H), 6.90 (d, $J = 8.8$ Hz, 2H), 6.10 (q, $J = 6.5$ Hz, 1H), 3.99 (t, $J = 6.5$ Hz, 2H), 1.87 – 1.73 (m, 2H), 1.65 (d, $J = 6.6$ Hz, 3H), 1.46 – 1.43 (m, 2H), 1.42 - 1.26 (m, 17H), 0.88 (t, $J = 6.7$ Hz, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 165.59, 162.96, 142.05, 131.62, 128.48, 127.74, 125.99, 122.65, 114.02, 72.44, 68.19, 31.90, 29.63, 29.61, 29.57, 29.54, 29.34, 29.09, 25.96, 22.67, 22.46, 14.10. HRMS: Calcd. for C$_{27}$H$_{39}$O$_3$ [M+H]$^+$ 411.2899, found 411.2906.

(S)-Octan-2-yl 4-(dodecyloxy)benzoate (2e):

\[
\text{C}_{12}\text{H}_{25}\text{O} = \text{O} = \text{O}
\]

Yield: 89%, colorless oil. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.97 (d, $J = 8.1$ Hz, 1H), 6.89 (d, $J = 8.0$ Hz, 1H), 5.08 – 5.14 (m, 1H), 4.00 (t, $J = 6.48$, 1H), 1.26 – 1.83 (m, 18H), 0.88 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 166.03, 162.80, 131.45, 123.15, 113.98, 71.25, 68.19, 36.14, 31.92, 31.75,

5α-Cholestan-3α-yl 4-(dodecyloxy)benzoate (2f):

Yield: 92%, white wax. $^1$H NMR (400 MHz, CDCl$_3$) δ 8.00 (d, $J$ = 8.8 Hz, 2H), 6.91 (d, $J$ = 8.9 Hz, 2H), 5.24 (s, 1H), 4.01 (t, $J$ = 6.6 Hz, 2H), 1.98 (dt, $J$ = 12.1, 2.8 Hz, 1H), 1.91 – 0.71 (m, 70H), 0.66 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 165.72, 162.78, 131.47, 123.39, 114.02, 70.25, 68.20, 56.57, 56.35, 54.42, 42.60, 40.45, 40.04, 39.51, 36.19, 35.87, 35.81, 35.49, 33.26, 33.08, 31.97, 31.91, 29.64, 29.62, 29.58, 29.55, 29.36, 29.34, 29.11, 28.43, 28.25, 28.00, 26.36, 25.98, 24.16, 23.86, 22.80, 22.68, 22.55, 20.84, 18.67, 14.10, 12.08, 11.41. HRMS: Calcd. for C$_{46}$H$_{77}$O$_{3}$ [M+H]^{+} 677.5873, found 677.5858.

(1S,2S,5R)-2-isopropyl-5-methylcyclohexyl 4-(dodecyloxy)benzoate (2g):

Yield: 17%, yellow oil. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.98 (d, $J$ = 8.8 Hz, 2H), 6.90 (d, $J$ = 8.8 Hz, 2H), 5.42 (s, 1H), 4.00 (t, $J$ = 6.5 Hz, 2H), 2.07 (ddd, $J$ = 14.2, 5.2, 3.3 Hz, 1H), 1.88 – 1.74 (m, 4H), 1.56 – 1.40 (m, 4H), 1.40 – 1.20 (m, 17H), 1.19 – 0.94 (m, 3H), 0.80 – 0.95 (m, 12H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 165.69, 162.82, 131.45, 123.26, 114.06, 71.25, 68.21, 47.08, 39.33, 34.93, 31.90, 29.63, 29.61, 29.57, 29.54, 29.41, 29.33, 29.09, 26.77, 25.97, 25.43, 22.67, 22.18, 20.94, 20.82, 14.09. HRMS: Calcd. for C$_{36}$H$_{49}$O$_{3}$ [M+H]^{+} 445.3682, found 445.3678.

(1S,2S)-2-benzyloxy-1-benzyloxymethyl-4-(tert-butyl-diphenyl-silanyloxy)butyl 4-(dodecyloxy)benzoate (2h):

Yield: 27%, colorless oil. $^1$H NMR (400 MHz, CDCl$_3$) δ 8.00 (d, $J$ = 8.5 Hz, 2H), 7.65 – 7.57 (m,
4H), 7.50 – 7.11 (m, 16H), 6.89 (d, J = 8.6 Hz, 2H), 5.46 – 5.44 (m, 1H), 4.71 – 4.45 (m, 4H), 4.12 – 4.09 (m, 1H), 4.00 (t, J = 6.4 Hz, 2H), 3.78 – 3.71 (m, 4H), 1.90 – 1.68 (m, 4H), 1.26 – 1.46 (m, 20H), 1.02 (s, 9H), 0.87 (t, J = 6.7 Hz, 3H). 13C NMR (100 MHz, CDCl3) δ 165.84, 163.07, 138.53, 138.17, 135.56, 133.84, 133.73, 131.84, 129.57, 128.34, 128.27, 127.85, 127.64, 127.58, 122.40, 114.08, 74.65, 73.73, 73.15, 73.09, 68.48, 68.26, 60.17, 33.59, 31.92, 29.64, 29.38, 29.14, 26.86, 26.00, 22.69, 19.15, 14.11. HRMS: Calcd. for C54H71O6Si [M+H]+ 843.5020, found 843.5034.

4,6-O-Benzylidene-1,2-dideoxy-3-O-4-(dodecyloxy)benzoyl-l-nitro-D-ribo–hex-l-enopyranose (2i):

Yield: 15%, white wax. 1H NMR (400 MHz, CDCl3) δ 8.00 (d, J = 8.7 Hz, 2H), 7.40-7.30 (m, 5H), 6.91 (d, J = 8.7 Hz, 2H), 6.59 (d, J = 6.0 Hz, 1H), 5.92 (dd, J = 5.3, 4.4 Hz, 1H), 5.66(s, 1H), 4.70 (dd, J = 10.5, 5.1 Hz, 1H), 4.60 (dt, J = 10.2, 5.5 Hz, 1H), 4.14 (dd, J = 10.3, 3.8 Hz, 1H), 4.07 (t, J = 10.5 Hz, 1H), 4.01 (t, J = 6.4 Hz, 2H), 1.81 – 1.78 (m, 2H), 1.46 – 1.42 (m, 2H), 1.35 – 1.26 (m, 20H), 0.88 (t, J = 6.4 Hz, 3H). 13C NMR (101 MHz, CDCl3) δ 165.23, 163.50, 154.65, 136.22, 131.91, 129.38, 128.33, 126.03, 121.16, 114.26, 101.95, 98.18, 74.75, 68.30, 68.21, 67.90, 61.58, 31.90, 29.63, 29.61, 29.56, 29.53, 29.33, 29.06, 25.95, 22.67, 14.10. HRMS: Calcd. for C32H42NO8 [M+H]+ 568.2905, found 568.2877.

4-(Dodecyloxy)benzoic anhydride (4):

Yield: 0-60%, white solids, m.p.: 56 – 57 °C. 1H NMR (400 MHz, CDCl3) δ 8.08 (d, J = 8.9 Hz, 4H), 6.96 (d, J = 8.9 Hz, 4H), 4.04 (t, J = 6.6 Hz, 4H), 1.86 – 1.76 (m, 4H), 1.52 – 1.41 (m, 4H), 1.41 – 1.20 (m, 34H), 0.88 (t, J = 6.8 Hz, 6H). 13C NMR (100 MHz, CDCl3) δ 164.19, 162.35, 132.79, 120.99, 114.53, 68.41, 31.88, 29.60, 29.55, 29.52, 29.31, 29.02, 25.93, 22.66, 14.08. HRMS: Calcd for C38H59O5 [M+H]+ 595.4357, found 595.4369. The data are identical with those reported in the literature.4

N,N’-Di-(4-(dodecyloxy))benzoyl -N,N’-bis(ethylcarboxy)hydrazine (5):
Yield: 0-49%, white solids, m.p.: 89 – 90 °C. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.72 (d, $J$ = 8.4 Hz, 4H), 6.91 (d, $J$ = 8.4 Hz, 4H), 4.20 (q, $J$ = 6.8 Hz, 4H), 4.00 (t, $J$ = 6.4, 4H), 1.82 – 1.76 (m, 4H), 1.59-1.27 (m, 36H), 1.15 (t, $J$ = 7.2 Hz, 6H), 0.88 (t, $J$ = 6.8 Hz, 6H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 168.62, 162.74, 152.90, 130.98, 125.94, 113.93, 68.22, 63.93, 31.89, 29.63, 29.60, 29.56, 29.53, 29.33, 29.07, 25.96, 22.66, 14.09, 13.87. HRMS: Calcd for C$_{44}$H$_{69}$N$_2$O$_8$ [M+H]$^+$ 753.5054, found 753.5045.

3-Butyn-2-yl 4-(octadecylsulfonyl)benzoate (8c):

Yield: 93%, colorless oil. $^1$H NMR (400 MHz, CDCl$_3$) δ 8.26 (d, $J$ = 8.4 Hz, 2H), 8.00 (d, $J$ = 8.4 Hz, 2H), 5.71 (qd, $J$ = 6.7, 2.0 Hz, 1H), 3.25 – 3.01 (m, 2H), 2.54 (d, $J$ = 2.0 Hz, 1H), 1.70-1.66 (m, 2H), 1.68 (d, $J$ = 6.8 Hz, 3H), 1.41 – 1.19 (m, 30H), 0.88 (t, $J$ = 6.7 Hz, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 163.90, 143.28, 134.47, 130.59, 128.23, 81.52, 73.66, 61.60, 56.24, 31.93, 29.69, 29.66, 29.55, 29.46, 29.36, 29.22, 28.99, 28.26, 22.70, 22.61, 21.28, 14.13. HRMS: Calcd. for C$_{29}$H$_{50}$NO$_4$S [M+NH$_4$]$^+$ 508.3461, found 508.3445.

1-Phenylethyl 4-(octadecylsulfonyl)benzoate (8d):

Yield: 93%, colorless oil. $^1$H NMR (400 MHz, CDCl$_3$) δ 8.26 (d, $J$ = 8.2 Hz, 2H), 7.99 (d, $J$ = 8.2 Hz, 2H), 7.46 – 7.31 (m, 5H), 6.17 (q, $J$ = 6.4 Hz, 1H), 3.11 – 3.07 (m, 2H), 1.72 (d, $J$ = 6.5 Hz, 3H), 1.71 – 1.69 (m, 2H), 1.18 – 1.40 (m, 30H), 0.88 (t, $J$ = 6.5 Hz, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 164.22, 142.93, 141.03, 135.15, 130.42, 128.65, 128.21, 128.15, 126.10, 73.99, 56.21, 31.90, 29.66, 29.52, 29.42, 29.34, 29.19, 28.95, 28.23, 22.67, 22.59, 22.19, 14.10. HRMS: Calcd. for C$_{33}$H$_{54}$NO$_4$S [M+NH$_4$]$^+$ 560.3773, found 560.3792.

(S)-octan-2-yl 4-(octadecylsulfonyl)benzoate (8e):
5α-Cholestan-3α-yl 4-(octadecylsulfonyl)benzoate (8f):

Yield: 96%, white wax. $^1$H NMR (400 MHz, CDCl$_3$) δ 8.24 (d, $J$ = 7.8 Hz, 2H), 8.01 (d, $J$ = 7.8 Hz, 2H), 5.32 (s, 1H), 3.19 – 3.00 (m, 2H), 1.99 (d, $J$ = 12.3 Hz, 2H), 1.90 – 0.78 (m, 76H), 0.67 (s, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 164.37, 142.77, 135.85, 130.35, 128.20, 72.03, 56.54, 56.34, 56.28, 54.41, 42.61, 40.56, 40.00, 39.52, 36.18, 35.91, 35.83, 35.48, 33.22, 32.96, 31.94, 29.70, 29.56, 29.46, 29.37, 29.24, 29.00, 28.40, 28.29, 28.02, 26.27, 24.17, 23.87, 22.83, 22.70, 22.65, 22.57, 20.86, 18.69, 14.14, 12.10, 11.42. HRMS: Calcd for C$_{52}$H$_{92}$NO$_4$S [M+NH$_4$]$^+$ 826.6747, found 826.6690.

(1S,2S,5R)-2-isopropyl-5-methylcyclohexyl 4-(octadecylsulfonyl)benzoate (8g):

Yield: 88%, white solids, m.p.: 56-57 °C. $^1$H NMR (400 MHz, CDCl$_3$) δ 8.22 (d, $J$ = 8.4 Hz, 2H), 7.99 (d, $J$ = 8.4 Hz, 2H), 5.50 (s, 1H), 3.12 – 3.08 (m, 2H), 2.11 (ddd, $J$ = 14.3, 5.6, 3.4, 1H), 1.93 – 1.79 (m, 2H), 1.77 – 1.65 (m, 3H), 1.58 – 0.83 (m, 47H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 164.29,

(2S,3R)-1,3-bis(benzyloxy)-5-((tert-butyldiphenylsilyl)oxy)pentan-2-yl 4-(octadecylsulfonyl)benzoate (8h):

\[
\text{TBDPSO} - \text{OBn} - \text{OBn} - \text{S} - \text{O} - \text{O} - \text{C}_{18}\text{H}_{37} \\
\text{O} - \text{O} - \text{Yield: 94%, colorless oil.} \quad ^1\text{H NMR (400 MHz, CDCl}_3\text{)} \delta 8.20 (d, J = 8.5 Hz, 2H), 7.96 (d, J = 8.5 Hz, 2H), 7.62 – 7.59 (m, 4H), 7.41 – 7.19 (m, 16H), 5.51 (dd, J = 10.4, 4.6 Hz, 1H), 4.62 (d, J = 11.7 Hz, 1H), 4.59 (d, J = 11.4 Hz, 1H), 4.57 (d, J = 12.1 Hz, 1H), 4.51 (d, J = 12.2 Hz, 1H), 4.12 (dt, J = 8.6, 4.4 Hz, 1H), 3.70 – 3.80 (m, 4H), 3.15 – 3.02 (m, 2H), 1.90 -1.81 (m, 1H), 1.77– 1.62 (m, 3H), 1.62 – 1.16 (m, 30H), 1.00 (s, 9H), 0.88 (t, J = 6.8 Hz, 3H). \quad ^{13}\text{C NMR (100 MHz, CDCl}_3\text{)} \delta 164.56, 143.03, 138.24, 137.87, 135.55, 134.92, 133.64, 133.56, 130.58, 129.70, 128.43, 128.35, 128.15, 127.81, 127.75, 127.70, 127.64, 75.12, 74.42, 73.22, 73.16, 68.29, 59.90, 56.29, 33.55, 31.95, 29.71, 29.68, 29.65, 29.58, 29.49, 29.38, 29.28, 29.04, 28.32, 26.86, 22.71, 22.67, 19.15, 14.15. HRMS: Calcd. for C_{60}H_{83}O_{7}SSi [M+H]^+ 975.5623, found 975.5631.

4,6-O-Benzylidene-1,2-dideoxy-3-O-4-(octadecylsulfonyl) benzoyl-1-nitro-D-ribo-hex-1-enopyranose (8i):

\[
\text{Ph} - \text{O} - \text{O} - \text{S} - \text{C}_{18}\text{H}_{37} \\
\text{Yield: 66%, white solids, m.p.: 93-95 °C.} \quad ^1\text{H NMR (400 MHz, CDCl}_3\text{)} \delta 8.23 (d, J = 8.2 Hz, 2H), 8.00 (d, J = 8.2 Hz, 2H), 7.39 – 7.31 (m, 5H), 6.59 (d, J = 6.0 Hz, 1H), 5.98 (dd, J = 5.6, 4.4 Hz, 1H), 5.69 (s, 1H), 4.72 (dd, J = 10.7, 5.2 Hz, 1H), 4.59 (td, J = 10.3, 5.3 Hz, 1H), 4.20 (dd, J = 10.5, 4.0 Hz, 1H), 4.09 (t, J = 10.5 Hz, 1H), 3.13 – 3.05 (m, 2H), 1.72 – 1.65 (m, 2H), 1.37 – 1.21 (m, 30H), 0.88 (t, J = 6.7 Hz, 3H). \quad ^{13}\text{C NMR (100 MHz, CDCl}_3\text{)} \delta 164.04, 154.95, 143.62, 136.06, 133.87, 130.62, 129.54, 128.40, 128.38, 125.94, 102.00, 97.17, 74.50, 68.28, 67.82, 62.80, 56.22, 31.93, 29.70, 29.67, 29.65, 29.57, 29.48, 29.37, 29.25, 28.02, 28.28, 22.70, 22.60, 14.14. HRMS:
Calcd. for C_{38}H_{54}NO_{9}S [M+H]^{+} 700.3519, found 700.3530.

3-Butyn-2-yl 4-(pentatriacontan-18-ylsulfonyl)benzoate (14c):

\[
\text{Yield: 94\%, colorless oil.} \quad ^1\text{H NMR (400 MHz, CDCl}_3) \delta 8.25 (d, J = 8.3 \text{ Hz, 2H}), 7.97 (d, J = 8.3 \text{ Hz, 2H}), 5.73 - 5.69 (m, 1H), 2.92 (m, 1H), 2.52 (d, J = 2.0 \text{ Hz, 1H}), 1.83 - 1.76 (m, 2H), 1.68 (d, J = 6.8 \text{ Hz, 3H}), 1.59-1.23 (m, 62H), 0.88 (t, J = 6.7 \text{ Hz, 6H}). \quad ^{13}\text{C NMR (100 MHz, CDCl}_3) \delta 163.93, 142.56, 134.29, 130.33, 128.92, 81.54, 73.61, 64.65, 61.55, 31.91, 29.68, 29.58, 29.48, 29.35, 29.23, 27.82, 26.71, 22.67, 21.27, 14.09. \quad \text{MS (MALDI) Calcd. for C}_{46}\text{H}_{80}\text{NaO}_{4}\text{S 751.6, found 751.3. Elemental Analysis: Calcd. for C}_{46}\text{H}_{80}\text{O}_{4}\text{S C 75.77\%, H 11.06\%; found C 75.67\%, H 11.03\%.}
\]

1-Phenylethyl 4-(pentatriacontan-18-ylsulfonyl)benzoate (14d):

\[
\text{Yield: 91\%, colorless oil.} \quad ^1\text{H NMR (400 MHz, CDCl}_3) \delta 8.24 (d, J = 8.3 \text{ Hz, 2H}), 7.96 (d, J = 8.3 \text{ Hz, 2H}), 7.38 (m, 5H), 6.16 (q, J = 6.5 \text{ Hz, 1H}), 2.90 (brs, 1H), 1.82 - 1.75 (m, 2H), 1.71 (d, J = 6.6 \text{ Hz, 3H}), 1.60 - 1.22 (m, 62H), 1.22 (t, J = 6.5 \text{ Hz, 6H}). \quad ^{13}\text{C NMR (100 MHz, CDCl}_3) \delta 164.32, 142.29, 141.12, 135.04, 130.20, 128.90, 128.67, 128.22, 126.14, 73.99, 64.68, 31.93, 29.70, 29.58, 29.49, 29.36, 29.24, 27.86, 26.74, 22.69, 22.20, 14.10. \quad \text{MS (MALDI) Calcd. for C}_{50}\text{H}_{84}\text{NaO}_{4}\text{S 803.6, found 803.7. Elemental Analysis: Calcd. for C}_{50}\text{H}_{84}\text{O}_{4}\text{S C 76.87\%, H 10.84\%; found C 76.67\%, H 10.73\%.}
\]

5a-Cholestan-3a-yl 4-(pentatriacontan-18-ylsulfonyl)benzoate (14f):

\[
\text{Yield: 95\%, colorless oil.} \quad ^1\text{H NMR (400 MHz, CDCl}_3) \delta 8.23 (d, J = 7.9 \text{ Hz, 2H}), 7.98 (d, J = 7.9 \text{ Hz, 2H}), 5.32 (s, 1H), 2.92 (s, 1H), 1.99 (d, J = 11.8 \text{ Hz, 1H}), 1.96 - 0.75 (m, 112H), 0.67 (s, 3H). \quad ^{13}\text{C NMR (100 MHz, CDCl}_3) \delta 164.41, 142.09, 135.68, 130.08, 128.91, 71.98, 64.67, 56.55, 56.39, 54.44, 42.63, 40.58, 40.03, 39.52, 36.20, 35.92, 35.82, 35.51, 33.24, 32.99, 31.92, 29.69, 29.66,
29.63, 29.58, 29.48, 29.35, 29.24, 28.42, 28.23, 28.00, 27.88, 26.76, 26.28, 24.17, 23.89, 22.79, 22.67, 22.54, 20.87, 18.68, 14.08, 12.09, 11.40. MS (MALDI) Calcd. for C_{69}H_{122}NaO_{4}S 1069.9, found 1070.1. Elemental Analysis: Calcd. for C_{69}H_{124}O_{4}S C 78.94%, H 11.91%; found C 78.73%, H 11.90%.

(1S,2S,5R)-2-Isopropyl-5-methylcyclohexyl 4-(pentatriacontan-18-ylsulfonyl) benzoate (14g):

\[
\text{C}_{16}H_{33}^+ \text{SO} \text{O} \text{C} \text{O} \text{C}_{16}H_{33}^-
\]

Yield: 80%, colorless oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.21 (d, $J = 8.0$ Hz, 2H), 7.97 (d, $J = 8.1$ Hz, 2H), 5.50 (s, 1H), 2.92 (s, 1H), 2.09 (d, $J = 14.3$ Hz, 1H), 1.91 – 1.76 (m, 4H), 1.69 (s, 1H), 1.66 – 0.74 (m, 82H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 164.38, 142.04, 135.46, 130.03, 128.93, 72.92, 64.61, 46.96, 39.12, 34.74, 31.91, 29.68, 29.57, 29.48, 29.40, 29.35, 29.24, 27.81, 26.80, 26.71, 25.37, 22.67, 22.11, 20.93, 20.77, 14.10. MS (MALDI) Calcd. for C$_{52}$H$_{94}$NaO$_4$S 837.7; found 837.8. Elemental Analysis: Calcd. for C$_{52}$H$_{94}$O$_4$S C 76.60%, H 11.62%; found C 76.44%, H 11.39%.

(1S,2S)-2-Benzyloxy-1-benzyloxymethyl-4-(tert-butyl-diphenyl-silanyloxy)-butyl 4-(pentatriacontan-18-ylsulfonyl)benzoate (14h):

\[
\text{C}_{16}H_{33}^+ \text{SO} \text{O} \text{C} \text{O} \text{C}_{16}H_{33}^-
\]

Yield: 94%, colorless oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.18 (d, $J = 8.2$ Hz, 2H), 7.93 (d, $J = 8.2$ Hz, 2H), 7.60 (d, $J = 6.2$ Hz, 4H), 7.45 – 7.18 (m, 16H), 5.50 (dd, $J = 9.4$, 4.8 Hz, 1H), 4.59 (t, $J = 12.6$ Hz, 1H), 4.57 (d, $J = 12.1$ Hz, 1H), 4.50 (d, $J = 12.1$ Hz, 1H), 4.12 (td, $J = 7.9$, 4.2 Hz, 1H), 3.73 – 3.78 (m, 4H), 2.91 (brs, 1H), 1.94 – 1.67 (m, 5H), 1.56 (m, 3H), 1.50 – 1.12 (m, 64H), 1.00 (s, 9H), 0.88 (t, $J = 6.5$ Hz, 6H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 164.59, 142.23, 138.23, 137.85, 135.51, 134.72, 133.64, 133.54, 130.30, 129.65, 128.38, 128.30, 127.77, 127.65, 127.59, 75.05, 74.43, 73.19, 73.12, 68.26, 64.68, 59.91, 33.54, 31.91, 29.68, 29.58, 29.51, 29.34, 29.27, 27.89, 26.84, 26.75, 22.67, 19.12, 14.08. MS (MALDI) Calcd. for C$_{77}$H$_{116}$NaO$_4$Si 1235.8; found
1235.6. Elemental Analysis: Calcd. for C_{77}H_{116}O_{7}SSi C 76.19%, H 9.63%; found C 76.09%, H 9.53%.

4,6-O-Benzylidene-1,2-dideoxy-3-O-4-(pentatriacontan-18-ylsulfonyl)benzoyl-1-nitro-D-ribo-hex-1-enopyranose (14i):

Yield: 65%, colorless oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.22 (d, $J = 7.9$ Hz, 1H), 7.97 (d, $J = 7.9$ Hz, 2H), 7.39 – 7.31 (m, 5H), 6.59 (d, $J = 5.8$ Hz, 1H), 6.11 – 5.86 (m, 1H), 5.69 (s, 1H), 4.72 (dd, $J = 10.5$, 5.1 Hz, 1H), 4.59 (td, $J = 10.3$, 5.4 Hz, 1H), 4.19 (dd, $J = 10.4$, 3.4 Hz, 1H), 4.09 (t, $J = 10.4$ Hz, 1H), 2.92 (s, 1H), 1.81 – 1.75 (m, 2H), 1.58 – 1.55 (m, 2H), 1.48 – 1.07 (m, 60H), 0.88 (t, $J = 6.2$ Hz, 6H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 164.06, 154.91, 142.83, 136.03, 133.65, 130.34, 129.50, 129.05, 128.35, 125.91, 102.00, 97.13, 74.48, 68.25, 67.79, 64.60, 62.76, 31.89, 29.67, 29.57, 29.48, 29.33, 29.23, 27.77, 26.67, 22.66, 14.09. MS (MALDI) Calcd. for C$_{55}$H$_{87}$NNaO$_9$S (M+Na) 960.6, found 960.7. Elemental Analysis: Calcd. for C$_{55}$H$_{87}$NO$_9$S C 70.40%, H 9.35%; found C 70.16%, H 9.41%.

**IV. The release of alcohols and recovery of the tagged acids**

**Take the hydrolysis of 14f to afford 15 as an example:**

To a solution of 14f (0.200 g, 0.19 mmol) in THF/methanol/H$_2$O (v/v/8/2/1, 10 mL) was added KOH (48 mg, 0.86 mmol), the mixture was stirred overnight and acidified with 1 M HCl. The solvent was removed and the residue was extracted with methylene dichloride (30 mL×3). The combined organic layer was dried over sodium sulfate and concentrated, followed by purification on silica gel (petroleum ether /ethyl acetate = 10/1 to 1/4) afforded white wax 5α-cholestan-3α-ol (15, 68 mg, 92%) and acid 13 (116 mg, 90%).
Data for 5α-cholestan-3α-ol (15): ^5,6^ H NMR (400 MHz, CDCl₃) δ 4.04 (s, 1H), 1.98 – 0.65 (m, 46H). ^13^C NMR (100 MHz, CDCl₃) δ 66.61, 56.52, 56.21, 54.31, 42.56, 40.02, 39.50, 39.11, 36.15, 36.05, 35.88, 35.79, 35.47, 32.16, 32.00, 29.00, 28.57, 28.23, 28.00, 24.16, 23.81, 22.81, 22.55, 20.76, 18.65, 12.06, 11.16.
V. NMR spectra

\[ \text{\textsuperscript{1}H NMR of S1} \]
\[ \text{\textsuperscript{13}C NMR of S1} \]
$^1$H NMR of S2
$^{13}$C NMR of S2
\[ ^1H \text{NMR of 7} \]
$^{13}$C NMR of 7
$^1$H NMR of S3
$^1$H NMR of 10
$^{13}$C NMR of 10
\( ^1\text{H} \) NMR of \( \text{II} \)
$^{13}$C NMR of 11
$^1$H NMR of 12
$^{13}$C NMR of 12
$^1$H NMR of 13
$^{13}$C NMR of 13
\(^1\text{H NMR of 2a}\)
$^1$H NMR of 2b
$^{13}$C NMR of 2b
$^{1}H$ NMR of 2c
$^{13}$C NMR of 2c
$^1$H NMR of 2d
$^{13}$C NMR of 2d
$^1$H NMR of 2e
$^{13}$C NMR of 2e
$^1$H NMR of 2f
$^{13}$C NMR of 2f
$^{1}H$ NMR of $2g$
$^{13}$C NMR of 2g
S46

$^1$H NMR of 2h
$^{13}$C NMR of 2h
$^1$H NMR of 2i
$^{13}$C NMR of 2i
$^1$H NMR of 4
\(^{13}\text{C} \text{NMR of 4}\)
$^1$H NMR of 5
$^{13}$C NMR of 5
$^1$H NMR of $8c$
$^{13}$C NMR of 8c
$^{13}$C NMR of 8d
$^1$H NMR of 8e
$^{13}$C NMR of 8e
$^{1}$$H$ NMR of 8f
$^{13}$C NMR of 8f
$^{1}H$ NMR of 8g
$\text{C}_{19}\text{H}_{37}$

$^{13}$C NMR of 8g
$^1$H NMR of 8h
$^{13}$C NMR of 8h
$^1$H NMR of 8i
$^{13}$C NMR of 8i
$^{1}$H NMR of 14c
$^{13}$C NMR of 14c
$^1$H NMR of 14d
$^{13}$C NMR of 14d
$^{1}H$ NMR of 14f
$^{13}$C NMR of 14f
$^1$H NMR of 14g
$^{13}$C NMR of 14g
$^1$H NMR of 14h
$^{13}$C NMR of 14h
$^{1}$H NMR of 14i
$^{13}$C NMR of $^{14i}$
$^1$H NMR of 15
$^{13}$C NMR of 15
VI. References


