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Characterization of compounds 13-53

1-Oleoyl-3-*O-tert*-butyldimethylsilyl-*sn*-glycerol 13. Acquired from 1-oleoyl-2,3-bis(trifluoroacetyl)-*sn*-glycerol (**5**; 1.097 g, 2.00 mmol) via **9**. Overall yield: 0.800 g (85%, colourless oil); R_f (pentane/toluene/EtOAc = 40:50:10, v/v/v) = 0.43; $[\alpha]_D^{20}$= +1.76 (*c* 9.45, $CHCl_3$); Found: C, 68.92; H, 11.50%. $C_{27}H_{54}O_4Si$ (470.80) requires C, 68.88; H, 11.56%. ^{1}H NMR δ_H (in ppm, $CDCl_3$, 400 MHz) 5.39-5.27 (2H, m, C*H*=C*H*); 4.19-4.06 (2H, m, RC(O)OCH_aH_bCHCH_2OSi); 3.87 (1H, tt, *J*=5.5, 5.5 Hz; C*H*OH); 3.67 (1H, dd, *J*=4.6, 4.6 Hz, RC(O)OCH_2CHC$H_a$$H_b$OSi); 3.60 (1H, dd, *J*=5.7, 5.5 Hz, RC(O)OCH_2CHC$H_a$$H_b$OSi); 2.33 (2H, t, *J*=7.5 Hz, 2-CH_2); 2.02 (4H, m, 8-CH_2, 11-CH_2); 1.62 (2H, m, 3-CH_2); 1.36-1.23 (20H, m, 4-7-CH_2, 12-17-CH_2); 0.86-0.90 (12H, m, 18-CH_3, *t-Bu*-Si); 0.07 (6H, s, CH_3-Si). ^{13}C NMR δ_C (in ppm, $CDCl_3$, 100 MHz) 174.14 (C1); 129.95, 130.22 (C9, C10); 34.39 (C2); 32.12 (C16); 29.32-29.98 (C4-C7, C12-C15); 27.38, 27.44 (C11, C8); 25.14 (C3); 22.90 (C17); 14.33 (C18): oleoyl fragment. 26.05 (*C*H_3-); 18.48 (*C*-Si); -5.26, -5.24 (*C*H_3-Si): *tert*-butyldimethylsilyl fragment; 70.22 (C2); 65.22 (C1); 63.91 (C3): glycerol fragment.

1-*O-tert*-Butyldimethylsilyl-3-oleoyl-*sn*-glycerol 14. Obtained from 3-oleoyl-1,2-bis(trifluoroacetyl)-*sn*-glycerol (**6**; 1.097 g, 2.00 mmol) via **10**. Overall yield: 0.772 g (82%, colourless oil); $[\alpha]_D^{20}$= -1.73 (*c* 9.63, $CHCl_3$). All other physicochemical and spectral characteristics were identical with those of **13**.

1-Oleoyl-3-*O*-triisopropylsilyl-*sn*-glycerol 15. Synthesized from 1-oleoyl-2,3-bis(trifluoroacetyl)-*sn*-glycerol (**5**; 1.097 g, 2.00 mmol) via **9**. Overall yield: 0.821 g (80%, colourless oil); R_f (pentane/toluene/EtOAc = 40:50:10, v/v/v) = 0.53; $[\alpha]_D^{20}$= +1.39 (*c* 13.36, $CHCl_3$); Found: C, 70.25; H, 11.63%. $C_{30}H_{60}O_4Si$ (512.88) requires C, 70.25; H, 11.79%. ^{1}H NMR δ_H (in ppm, $CDCl_3$, 400 MHz) 5.41-5.27 (2H, m, C*H*=C*H*); 4.15 (2H, m, RC(O)OCH_aH_bCHCH_2OSi); 3.89 (1H, tt, *J*=5.3, 5.3 Hz, C*H*OH); 3.76 (1H, dd, *J*=4.6, 4.6 Hz, RC(O)OCH_2CHC$H_a$$H_b$OSi); 3.70 (1H, dd, *J*=5.7, 5.7 Hz, RC(O)OCH_2CHC$H_a$$H_b$OSi); 2.33 (2H, t, *J*=7.5 Hz, 2-CH_2); 2.00 (4H, m, 8-CH_2, 11-CH_2); 1.62 (2H, m, 3-CH_2); 1.35-1.22 (20H, m, 4-7-CH_2, 12-17-CH_2); 1.00-1.16 (21H, m, CH_3C*H*-Si); 0.87 (3H, t, *J*=7.0 Hz,18-CH_3). ^{13}C NMR δ_C (in ppm, $CDCl_3$, 100 MHz) 174.13 (C1); 129.95, 130.22 (C9, C10); 34.41 (C2); 32.12 (C16); 29.32-29.98 (C4-C7, C12-C15); 27.38, 27.43 (C11, C8); 25.15 (C3); 22.90

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(C17); 14.32 (C18): oleoyl fragment; 18.13 (*C*H_3-);12.08 (*C*H-Si): triisopropylsilyl fragment; 70.33 (C2); 65.18 (C1); 64.22 (C3): glycerol fragment.

1-*O*-Triisopropylsilyl-3-oleoyl-*sn*-glycerol 16. Acquired from 3-oleoyl-1,2-bis(trifluoroacetyl)-*sn*-glycerol (**6**; 1.097 g, 2.00 mmol) via **10**. Overall yield: 0.851 g (83%, colourless oil); $[\alpha]_D^{20}$= -1.38 (*c* 10.17, $CHCl_3$). All other physicochemical and spectral characteristics were identical with those of **15**.

1-*O*-Hexadecyl-3-*O*-*tert*-butyldimethylsilyl-*sn*-glycerol 17. Prepared from 1-*O*-hexadecyl-2,3-bis(trifluoroacetyl)-*sn*-glycerol (**7**; 1.017 g, 2.00 mmol) via **11**. Overall yield: 0.732 g (85%, colourless oil); R_f (pentane/toluene/EtOAc = 40:50:10, v/v/v) = 0.47; $[\alpha]_D^{20}$= +0.40 (*c* 12.97, $CHCl_3$); Found: C, 69.93; H, 12.55%. $C_{25}H_{54}O_3Si$ (430.78) requires C, 69.70; H, 12.63%. ^{1}H NMR δ_H (in ppm, $CDCl_3$, 400 MHz) 3.80 (1H, tt, *J*=5.5, 5.3 C*H*OH); 3.70-3.57 (2H, m, $SiOCH_2CHCH_aH_bOR$); 3.50-3.48 (4H, m, 1-CH_2, $ROCH_2CHCH_aH_bOSi$); 1.60-1.52 (2H, m, 2-CH_2); 1.33-1.17 (26H, m, 3-15-CH_2); 0.83-0.93 (12H, m, 16-CH_3, *t-Bu*-Si); 0.06 (6H, s, CH_3-Si). ^{13}C NMR δ_C (in ppm, $CDCl_3$, 100 MHz) 71.88 (C1); 32.14 (C2); 29.57-29.91 (C4-C14); 26.34 (C3); 22.91 (C15); 14.33 (C16): hexadecyl fragment; 26.09 (*C*H_3-);18.51 (*C*-Si); -5.20 (*C*H_3-Si): *tert*-butyldimethylsilyl fragment; 71.62 (C1); 70.86 (C2); 64.28 (C3): glycerol fragment.

1-*O*-*tert*-Butyldimethylsilyl-3-*O*-hexadecyl-*sn*-glycerol 18. Synthesized from 3-*O*-hexadecyl-1,2-bis(trifluoroacetyl)-*sn*-glycerol (**8**; 1.017 g, 2.00 mmol) via **12**. Overall yield: 0.735 g (85%, colourless oil); $[\alpha]_D^{20}$= -0.49 (*c* 8.66, $CHCl_3$). All other physicochemical and spectral characteristics were identical with those of **17**.

1-Oleoyl-2-acetyl-3-*O*-*tert*-butyldimethylsilyl-*sn*-glycerol 19. Obtained from 1-oleoyl-3-*O*-*tert*-butyldimethylsilyl-*sn*-glycerol (**13**; 0.706 g, 1.50 mmol) and acetyl chloride (0.161 mL, 2.25 mmol). Yield: 0.723 g (94%, colourless oil); R_f (pentane/toluene/EtOAc = 40:50:10, v/v/v) = 0.62; $[\alpha]_D^{20}$= +9.75 (*c* 11.05, $CHCl_3$); Found: C, 67.88; H, 11.08%. $C_{29}H_{56}O_5Si$ (512.84) requires C, 67.92; H, 11.01%. ^{1}H NMR δ_H (in ppm, $CDCl_3$, 400 MHz) 5.39-5.29 (2H, m, C*H*=C*H*); 5.05 (1H, m, C*H*OC(O)); 4.34 (1H, dd, *J*=3.7, 3.7 Hz, $RC(O)OCH_aH_bCHCH_2OSi$); 4.16 (1H, dd, *J*=6.0, 6.2 Hz, $RC(O)OCH_aH_bCHCH_2OSi$); 3.71 (2H, d, *J*=5.3 Hz, $RC(O)OCH_2CHCH_aH_bOSi$); 2.30 (2H, t, *J*=7.5 Hz, 2-CH_2); 2.06 (3H, s, 2-CH_3);

2.06-1.98 (4H, m, 8-CH_2, 11-CH_2); 1.66-1.58 (2H, m, 3-CH_2); 1.37-1.23 (20H, m, 4-7-CH_2, 12-17-CH_2); 0.86-0.90 (12H, m, 18-CH_3, *t-Bu*-Si); 0.05 (6H, s, CH_3-Si). ^{13}C NMR δ_C (in ppm, $CDCl_3$, 100 MHz) 173.67 (C1); 129.95, 130.23 (C9, C10); 34.36 (C2); 32.12 (C16); 29.30-29.98 (C4-C7, C12-C15); 27.39, 27.44 (C11, C8); 25.11 (C3); 22.90 (C17); 14.33 (C18): oleoyl fragment; 170.50 (C1); 21.22 (C2): acetyl fragment; 25.96 (*C*H_3-);18.42 (*C*-Si); -5.29, -5.26 (*C*H_3-Si): *tert*-butyldimethylsilyl fragment; 72.20 (C2); 62.58 (C1); 61.62 (C3): glycerol fragment.

1-*O*-*tert*-Butyldimethylsilyl-2-acetyl-3-oleoyl-*sn*-glycerol 20. Acquired from 1-*O*-*tert*-butyldimethylsilyl-3-oleoyl-*sn*-glycerol (**14**; 0.706 g, 1.50 mmol) and acetyl chloride (0.161 mL, 2.25 mmol). Yield: 0.731 g (95%, colourless oil); $[\alpha]_D^{20}$= -9.47 (*c* 9.90, $CHCl_3$). All other physicochemical and spectral characteristics were identical with those of **19**.

1-Oleoyl-2-acetyl-3-*O*-triisopropylsilyl-*sn*-glycerol 21. Prepared from 1-oleoyl-3-*O*-triisopropylsilyl-*sn*-glycerol (**15**; 0.769 g, 1.50 mmol) and acetyl chloride (0.161 mL, 2.25 mmol). Yield: 0.766 g (92%, colourless oil); R_f (pentane/toluene/EtOAc = 40:50:10, v/v/v) = 0.61; $[\alpha]_D^{20}$= +11.28 (*c* 9.87, $CHCl_3$); Found: C, 69.20; H, 11.30%. $C_{32}H_{62}O_5Si$ (554.92) requires C, 69.26; H, 11.26%. ^{1}H NMR δ_H (in ppm, $CDCl_3$, 400 MHz) 5.41-5.29 (2H, m, C*H*=C*H*); 5.10-5.06 (1H, m, C*H*OC(O)); 4.37 (1H, dd, *J*=3.8, 3.8 Hz, RC(O)OC$H_a$$H_bCHCH_2$OSi); 4.19 (1H, dd, *J*=6.0, 6.0 Hz, RC(O)OC$H_a$$H_bCHCH_2$OSi); 3.81 (2H, d, *J*=5.2 Hz, RC(O)OCH_2CHC$H_a$$H_b$OSi); 2.30 (2H, t, *J*=7.4 Hz, 2-CH_2); 2.05 (3H, s, 2-CH_3); 2.03-1.97 (4H, m, 8-CH_2, 11-CH_2); 1.63-1.57 (2H, m, 3-CH_2); 1.35-1.21 (20H, m, 4-7-CH_2, 12-17-CH_2); 1.12-0.98 (21H, m, CH_3C*H*-Si); 0.87 (3H, t, *J*=7.1 Hz,18-CH_3). ^{13}C NMR δ_C (in ppm, $CDCl_3$, 100 MHz) 173.67 (C1); 129.94, 130.22 (C9, C10); 34.37 (C2); 32.12 (C16); 29.31-29.98 (C4-C7, C12-C15); 27.39, 27.43 (C11, C8); 25.12 (C3); 22.89 (C17); 14.31 (C18): oleoyl fragment; 170.51 (C1); 21.20 (C2): acetyl fragment; 18.07 (*C*H_3-);12.08 (*C*H-Si): triisopropylsilyl fragment; 72.30 (C2); 62.60 (C1); 62.01 (C3): glycerol fragment.

1-*O*-Triisopropylsilyl-2-acetyl-3-oleoyl-*sn*-glycerol 22. Synthesized from 1-*O*-triisopropylsilyl-3-oleoyl-*sn*-glycerol (**16**; 0.769 g, 1.50 mmol) and acetyl chloride (0.161 mL, 2.25 mmol). Yield: 0.774 g (93%, colourless oil); $[\alpha]_D^{20}$= -11.81

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(*c* 10.91, $CHCl_3$). All other physicochemical and spectral characteristics were identical with those of **21**.

1,2-Dioleoyl-3-*O*-triisopropylsilyl-*sn*-glycerol 23. Obtained from 1-oleoyl-3-*O*-triisopropylsilyl-*sn*-glycerol (**15**; 0.769 g, 1.50 mmol) and oleoyl chloride (0.744 mL, 2.25 mmol). Yield: 1.084 g (93%, colourless oil); R_f (pentane/toluene/EtOAc = 40:50:10, v/v/v) = 0.83; $[\alpha]_D^{20}$= +10.00 (*c* 10.12, $CHCl_3$); Found: C, 74.23; H, 11.88%. $C_{48}H_{92}O_5Si$ (777.33) requires C, 74.17; H, 11.93%. ^{1}H NMR δ_H (in ppm, $CDCl_3$, 400 MHz) 5.37-5.31 (4H, m, C*H*=C*H*); 6.01-5.07 (1H, m, C*H*OC(O)); 4.37 (1H, dd, *J*=3.8, 3.8 Hz, RC(O)OCH_aH$_b$CHCH$_2$OSi); 4.19 (1H, dd, *J*=6.3, 6.3 Hz, RC(O)OCH$_a$$H_b$CHCH$_2$OSi); 3.81 (2H, d, *J*=5.2 Hz, RC(O)OCH$_2$CHC$H_a$$H_b$OSi); 2.29 (4H, t, *J*=7.4 Hz, 2-CH_2); 2.00 (8H, m, 8-CH_2, 11-CH_2); 1.64-1.56 (4H, m, 3-CH_2); 1.40-1.20 (40H, m, 4-7-CH_2, 12-17-CH_2); 1.12-0.98 (21H, m, CH_3C*H*-Si); 0.87 (6H, t, *J*=6.9 Hz,18-CH_3). ^{13}C NMR δ_C (in ppm, $CDCl_3$, 100 MHz) 173.30, 173.64 (C1); 129.94, 130.22 (C9, C10); 34.38, 34.55 (C2); 32.12 (C16); 29.31-29.99 (C4-C7, C12-C15); 27.40, 27.44 (C11, C8); 25.13 (C3); 22.90 (C17); 14.32 (C18): oleoyl fragment; 18.09 (*C*H$_3$-); 12.09 (*C*H-Si): triisopropylsilyl fragment; 72.02 (C2); 62.69 (C1); 62.09 (C3): glycerol fragment.

1-*O*-Triisopropylsilyl-2-oleoyl-3-palmitoyl-*sn*-glycerol 24. Prepared in two steps by silylation of 3-palmitoyl-*sn*-glycerol (0.661 g, 2.00 mmol) with triisopropylchlorosilane (0.550 mL, 2.60 mmol) identically with **16** (see General procedure **3.3.**), followed by acylation of the intermediary 1-*O*-triisopropylsilyl-3-palmitoyl-*sn*-glycerol with oleoyl chloride (0.661 mL, 2.00 mmol), as described for **23**. Overall yield: 1.097 g (73%, colourless oil); R_f (pentane/toluene/EtOAc = 40:50:10, v/v/v) = 0.63; $[\alpha]_D^{20}$= -10.18 (*c* 9.31, $CHCl_3$); Found: C, 73.60; H, 12.00%. $C_{46}H_{90}O_5Si$ (751.29) requires C, 73.54; H, 12.07%. ^{1}H NMR δ_H (in ppm, $CDCl_3$, 400 MHz) 5.37-5.31 (2H, m, C*H*=C*H*); 5.09 (1H, m, C*H*OC(O)); 4.37 (1H, dd, *J*=3.7, 3.8 Hz, RC(O)OCH_aH$_b$CHCH$_2$OSi); 4.19 (1H, dd, *J*=6.2, 6.2 Hz, RC(O)OCH$_a$$H_b$CHCH$_2$OSi); 3.84-3.77 (2H, m, RC(O)OCH$_2$CHC$H_a$$H_b$OSi); 2.33-2.27 (4H, m, 2-C$H_{2,\ Palm}$, 2-CH_2); 2.02-1.98 (4H, m, 8-CH_2, 11-CH_2); 1.64-1.56 (4H, m, 3-C$H_{2,\ Palm}$, 3-CH_2); 1.22-1.38 (44H, m, 4-15-C$H_{2,\ Palm}$, 4-7-CH_2, 12-17-CH_2); 1.05 (21H, m, CH_3C*H*-Si); 0.88 (6H, t, *J*=7.1 Hz, 16-CH$_3$, 18-CH$_3$); ^{13}C NMR δ_C (in ppm, $CDCl_3$, 100 MHz) 173.32, 173.69 (C1); 129.94, 130.22 (C9, C10); 34.41,

34.55 (C2); 32.15 (C14); 32.12 (C16); 29.31-29.99 (C4-C13, C4-C7, C12-C15); 27.40, 27.44 (C11, C8); 25.14 (C3); 22.91 (C17); 14.33 (C16, C18): oleoyl and palmitoyl fragments; 18.10 (*C*H_3-);12.09 (*C*H-Si): triisopropylsilyl fragment; 72.02 (C2); 62.68 (C3); 62.08 (C1): glycerol fragment.

1-*O*-Hexadecyl-2-acetyl-3-*O*-*tert*-butyldimethylsilyl-*sn*-glycerol 25. Obtained from 1-*O*-hexadecyl-3-*O*-*tert*-butyldimethylsilyl-*sn*-glycerol (**17**; 0.646 g, 1.50 mmol) and acetyl chloride (0.161 mL, 2.25 mmol). Yield: 0.674 g (95%, colourless oil); R_f (pentane/toluene/EtOAc = 40:50:10, v/v/v) = 0.58; $[\alpha]_D^{20}$= +4.51 (*c* 14.60, $CHCl_3$); Found: C, 68.67; H, 11.87%. $C_{27}H_{56}O_4Si$ (472.82) requires C, 68.59; H, 11.94%. ^{1}H NMR δ_H (in ppm, $CDCl_3$, 400 MHz) 4.99 (1H, tt, *J*=5.1, 5.1 C*H*OC(O)); 3.75-3.67 (2H, m, $SiOCH_2CHCH_aH_bOR$); 3.57-3.53 (2H, m, $ROCH_2CHCH_aH_bOSi$); 3.45-3.37 (2H, m, 1-CH_2); 2.06 (3H, s, 2-CH_3); 1.57-1.51 (2H, m, 2-CH_2); 1.33-1.17 (26H, m, 3-15-CH_2); 0.82-0.86 (12H, m, 16-CH_3, *t-Bu*-Si); 0.05 (6H, s, CH_3-Si). ^{13}C NMR δ_C (in ppm, $CDCl_3$, 100 MHz) 71.83 (C1); 32.14 (C2); 29.57-29.91 (C4-C14); 26.28 (C3); 22.91 (C15); 14.33 (C16): hexadecyl fragment; 170.76 (C1); 21.40 (C2): acetyl fragment; 26.01 (*C*H_3-);18.44 (*C*-Si); -5.24, -5.22 (*C*H_3-Si): *tert*-butyldimethylsilyl fragment; 73.51 (C2); 69.07 (C1); 61.86 (C3): glycerol fragment.

1-*O*-*tert*-Butyldimethylsilyl-2-acetyl-3-*O*-hexadecyl-*sn*-glycerol 26. Acquired from 1-*O*-*tert*-butyldimethylsilyl-3-O-hexadecyl-*sn*-glycerol (**18**; 0.646 g, 1.50 mmol) and acetyl chloride (0.161 mL, 2.25 mmol). Yield: 0.659 g (93%, colourless oil); $[\alpha]_D^{20}$= -5.00 (c 8.83, $CHCl_3$). All other physicochemical and spectral characteristics were identical with those of **25**.

1-Oleoyl-2-trichloroacetyl-3-*O*-*tert*-butyldimethylsilyl-*sn*-glycerol 27. Synthesized from 1-oleoyl-3-*O*-*tert*-butyldimethylsilyl-*sn*-glycerol (**13**; 0.706 g, 1.50 mmol) and trichloroacetyl chloride (0.252 mL, 2.25 mmol). Yield: 0.868 g (94%, colourless oil); R_f (pentane/toluene/EtOAc = 40:50:10, v/v/v) = 0.70; $[\alpha]_D^{20}$= +11.87 (*c* 10.26, $CHCl_3$); Found: C, 56.67; H, 8.60; Cl, 17.30%. $C_{29}H_{53}Cl_3O_5Si$ (616.17) requires C, 56.53; H, 8.67; Cl, 17.26%. ^{1}H NMR δ_H (in ppm, $CDCl_3$, 400 MHz) 5.37-5.31 (2H, m, C*H*=C*H*); 5.24-5.18 (1H, m, C*H*OC(O)); 4.44 (1H, dd, *J*=3.3, 3.3 Hz, $RC(O)OCH_aH_bCHCH_2OSi$); 4.26 (1H, dd, *J*=7.1, 7.1 Hz,

RC(O)OCH$_a$*H$_b$*CHCH$_2$OSi); 3.83 (2H, d, *J*=5.1 Hz, RC(O)OCH$_2$CHC*H$_a$H$_b$*OSi); 2.30 (2H, t, *J*=7.5 Hz, 2-C*H$_2$*); 2.04-1.96 (4H, m, 8-C*H$_2$*, 11-C*H$_2$*); 1.62-1.58 (2H, m, 3-C*H$_2$*); 1.36-1.20 (20H, m, 4-7-C*H$_2$*, 12-17-C*H$_2$*); 0.86-0.90 (12H, m, 18-C*H$_3$*, *t-Bu*-Si); 0.07 (6H, d, *J*=1.8 Hz, C*H$_3$*-Si). ^{13}C NMR δ_C (in ppm, $CDCl_3$, 100 MHz) 173.47 (C1); 129.95, 130.23 (C9, C10); 34.26 (C2); 32.12 (C16); 29.30-29.98 (C4-C7, C12-C15); 27.38, 27.44 (C11, C8); 25.03 (C3); 22.90 (C17); 14.33 (C18): oleoyl fragment; 161.75 (C1): trichloroacetyl fragment; 25.91 (*C*H$_3$-);18.36 (*C*-Si); -5.37, -5.33 (*C*H$_3$-Si): *tert*-butyldimethylsilyl fragment; 77.69 (C2); 62.09 (C1); 61.30 (C3): glycerol fragment.

1-Oleoyl-2-acetyl-3-trichloroacetyl-*sn*-glycerol 28. Obtained from 1-oleoyl-2-acetyl-3-*O*-*tert*-butyldimethylsilyl-*sn*-glycerol (**19**; 0.513 g, 1.00 mmol). Yield: 0.506 g (93%, colourless oil); R_f (pentane/toluene/EtOAc = 40:50:10, v/v/v) = 0.56; $[\alpha]_D^{20}$= -0.40 (*c* 7.18, $CHCl_3$); Found: C, 55.00; H, 7.70; Cl, 19.73%. $C_{25}H_{41}Cl_3O_6$ (543.95) requires C, 55.20; H, 7.60; Cl, 19.55%. ^{1}H NMR δ_H (in ppm, $CDCl_3$, 400 MHz) 5.36-5.32 (3H, m, C*H*=C*H*, C*H*OC(O)); 4.59 (1H, dd, *J*=3.8, 3.8 Hz, RC(O)OCH$_2$CHCH$_a$*H$_b$*OC(O)CCl$_3$); 4.46 (1H, dd, *J*=5.9, 5.7 Hz, RC(O)OCH$_2$CHC*H$_a$*H$_b$OC(O)CCl$_3$); 4.35 (1H, dd, *J*=4.8, 4.8 Hz, RC(O)OC*H$_a$*H$_b$CHCH$_2$OC(O)CCl$_3$); 4.20 (1H, dd, *J*=5.5, 5.5 Hz, RC(O)OCH$_a$*H$_b$*CHCH$_2$OC(O)CCl$_3$); 2.33 (2H, t, *J*=7.5 Hz, 2-C*H$_2$*); 2.08 (3H, s, 2-C*H$_3$*); 2.05-1.98 (4H, m, 8-CH$_2$, 11-C*H$_2$*); 1.66-1.56 (2H, m, 3-C*H$_2$*); 1.39-1.21 (20H, m, 4-7-C*H$_2$*, 12-17-C*H$_2$*); 0.88 (3H, t, *J*= 6.7 Hz, 18-CH$_3$). ^{13}C NMR δ_C (in ppm, $CDCl_3$, 100 MHz) 173.36 (C1); 129.92, 130.25 (C9, C10); 34.19 (C2); 32.12 (C16); 29.28-29.98 (C4-C7, C12-C15); 27.37, 27.44 (C11, C8); 25.04 (C3); 22.90 (C17); 14.33 (C18): oleoyl fragment; 170.07 (C1); 20.95 (C2): acetyl fragment; 161.87 (C1): trichloroacetyl fragment; 68.63 (C2); 66.61 (C3); 61.67 (C1): glycerol fragment.

1-Trichloroacetyl-2-acetyl-3-oleoyl-*sn*-glycerol 29. Obtained from 1-*O*-triisopropylsilyl-2-acetyl-3-oleoyl-*sn*-glycerol (**22**; 0.555 g, 1.00 mmol). Yield: 0.489 g (90%, colourless oil); $[\alpha]_D^{20}$= +0.42 (*c* 9.47, $CHCl_3$). All other physicochemical and spectral characteristics were identical with those of **28**.

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1,2-Dioleoyl-3-trichloroacetyl-*sn*-glycerol 30. Obtained from 1,2-dioleoyl-3-*O*-triisopropylsilyl-*sn*-glycerol (**23**; 0.777 g, 1.00 mmol). Yield: 0.705 g (92%, colourless oil); R_f (pentane/toluene/EtOAc = 40:50:10, v/v/v) = 0.64; $[\alpha]_D^{20}$= -0.21 (*c* 5.33, $CHCl_3$); Found: C, 64.20; H, 9.40; Cl, 13.90%. $C_{41}H_{71}Cl_3O_6$ (766.36) requires C, 64.26; H, 9.34; Cl, 13.88%. ^{1}H NMR δ_H (in ppm, $CDCl_3$, 400 MHz) 5.37-5.29 (5H, m, C*H*=C*H*, C*H*OC(O)); 4.58 (1H, dd, *J*=4.1, 4.1 Hz, RC(O)OCH$_2$CHCH$_a$*H$_b$*OC(O)CCl$_3$); 4.44 (1H, dd, *J*=5.8, 5.8 Hz, RC(O)OCH$_2$CHC*H$_a$*H$_b$OC(O)CCl$_3$); 4.34 (1H, dd, *J*=4.9, 4.7 Hz, RC(O)OC*H$_a$*H$_b$CHCH$_2$OC(O)CCl$_3$); 4.19 (1H, dd, *J*=5.5, 5.5 Hz, RC(O)OCH$_a$*H$_b$*CHCH$_2$OC(O)CCl$_3$); 2.32 (4H, t, *J*=7.4 Hz, 2-C*H$_2$*); 2.00 (8H, m, 8-C*H$_2$*, 11-C*H$_2$*); 1.63-1.57 (4H, m, 3-C*H$_2$*); 1.37-1.21 (40H, m, 4-7-C*H$_2$*, 12-17-C*H$_2$*); 0.87 (6H, t, *J*= 6.9 Hz, 18-C*H$_3$*). ^{13}C NMR δ_C (in ppm, $CDCl_3$, 100 MHz) 172.85, 173.32 (C1); 129.90, 130.24 (C9, C10); 34.19, 34.28 (C2); 32.12 (C16); 29.25-29.98 (C4-C7, C12-C15); 27.38, 27.44 (C11, C8); 24.99, 25.04 (C3); 22.90 (C17); 14.32 (C18): oleoyl fragment; 161.85 (C1): trichloroacetyl fragment; 68.40 (C2); 66.73 (C3); 61.73 (C1): glycerol fragment.

1-*O*-Hexadecyl-2-acetyl-3-trichloroacetyl-*sn*-glycerol 31. Obtained from 1-*O*-hexadecyl-2-acetyl-3-*O*-*tert*-butyldimethylsilyl-*sn*-glycerol (**25**; 0.473 g, 1.00 mmol). Yield: 0.464 g (92%, colourless oil); R_f (pentane/toluene/EtOAc = 40:50:10, v/v/v) = 0.54; $[\alpha]_D^{20}$= -5.81 (*c* 11.81, $CHCl_3$); Found: C, 54.82; H, 8.27; Cl, 21.00%. $C_{23}H_{41}Cl_3O_5$ (503.93) requires C, 54.82; H, 8.20; Cl, 21.11%. ^{1}H NMR δ_H (in ppm, $CDCl_3$, 400 MHz) 5.29-5.25 (1H, m, C*H*OC(O)); 4.61 (1H, dd, *J*=3.5, 3.7 Hz, ROCH$_2$CHCH$_a$*H$_b$*OC(O)CCl$_3$); 4.50 (1H, dd, *J*=6.0, 6.0 Hz, ROCH$_2$CHC*H$_a$*H$_b$OC(O)CCl$_3$); 3.61-3.56 (2H, m, CCl$_3$C(O)OCH$_2$CHC*H$_a$H$_b$*OR); 3.47-3.43 (2H, m, 1-C*H$_2$*); 2.08 (3H, s, 2-C*H$_3$*); 1.57-1.53 (2H, m, 2-C*H$_2$*); 1.33-1.17 (26H, m, 3-15-C*H$_2$*); 0.87 (3H, t, *J*= 6.8 Hz, 16-C*H$_3$*). ^{13}C NMR δ_C (in ppm, $CDCl_3$, 100 MHz) 72.12 (C1); 32.14 (C2); 29.57-29.91 (C4-C14); 26.24 (C3); 22.91 (C15); 14.33 (C16): hexadecyl fragment; 170.32 (C1); 21.09 (C2): acetyl fragment; 161.95 (C1): trichloroacetyl fragment; 69.73 (C2); 68.37 (C1); 67.25 (C3): glycerol fragment.

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1-Trichloroacetyl-2-acetyl-3-*O*-hexadecyl-*sn*-glycerol 32. Obtained from 1-*O*-*tert*-butyldimethylsilyl-2-acetyl-3-*O*-hexadecyl-*sn*-glycerol (**26**; 0.473 g, 1.00 mmol). Yield: 0.470 g (93%, colourless oil); $[\alpha]_D^{20}$= +6.00 (*c* 5.44, $CHCl_3$). All other physicochemical and spectral characteristics were identical with those of **31**.

1-Oleoyl-2-trichloroacetyl-3-acetyl-*sn*-glycerol 33. Obtained from 1-oleoyl-2-trichloroacetyl-3-*O*-*tert*-butyldimethylsilyl-*sn*-glycerol (**27**; 0.616 g, 1.00 mmol) and acetic anhydride (0.284 mL, 3.00 mmol). Yield: 0.511 g (94%, colourless oil); R_f (pentane/toluene/EtOAc = 40:50:10, v/v/v) = 0.49; $[\alpha]_D^{20}$= -0.68 (*c* 9.77, $CHCl_3$); Found: C, 55.30; H, 7.55; Cl, 19.47%. $C_{25}H_{41}Cl_3O_6$ (543.95) requires C, 55.20; H, 7.60; Cl, 19.55%. ^{1}H NMR δ_H (in ppm, $CDCl_3$, 400 MHz) 5.28-5.44 (3H, mm, C*H*=C*H*, C*H*OC(O)); 4.37-4.45 (2H, m, RC(O)OCH_aH$_b$CHCH$_2$OC(O)CH$_3$, RC(O)OCH$_2$CHCH$_a$$H_b$OC(O)CH$_3$); 4.22-4.30 (2H, m, RC(O)OCH$_2$CHCH_aH$_b$OC(O)CH$_3$, RC(O)OCH$_a$$H_b$CHCH$_2$OC(O)CH$_3$); 2.32 (2H, t, *J*=7.3 Hz, 2-CH_2); 2.08 (3H, s, 2-CH_3); 2.03-1.97 (4H, m, 8-CH_2, 11-CH_2); 1.63-1.57 (2H, m, 3-CH_2); 1.38-1.22 (20H, m, 4-7-CH_2, 12-17-CH_2); 0.87 (3H, t, *J*=7.1 Hz, 18-CH$_3$). ^{13}C NMR δ_C (in ppm, $CDCl_3$, 100 MHz) 173.28 (C1); 129.93, 130.24 (C9, C10); 34.14 (C2); 32.12 (C16); 29.26-29.98 (C4-C7, C12-C15); 27.38, 27.44 (C11, C8); 24.98 (C3); 22.90 (C17); 14.33 (C18): oleoyl fragment; 170.48 (C1); 20.77 (C2): acetyl fragment; 161.57 (C1): trichloroacetyl fragment; 74.69 (C2); 61.81 (C3); 61.61 (C1): glycerol fragment;

1-Oleoyl-2-trichloroacetyl-3-palmitoyl-*sn*-glycerol 34. Obtained from 1-oleoyl-2-trichloroacetyl-3-*O*-*tert*-butyldimethylsilyl-*sn*-glycerol (**27**; 0.616 g, 1.00 mmol) and palmitic anhydride (1.484 g, 3.00 mmol). Yield: 0.711 g (96%, colourless oil); R_f (pentane/toluene/EtOAc = 40:50:10, v/v/v) = 0.58; $[\alpha]_D^{20}$= 0.00 (*c* 4.26, $CHCl_3$); Found: C, 63.35; H, 9.50; Cl, 14.30%. $C_{39}H_{69}Cl_3O_6$ (740.32) requires C, 63.27; H, 9.39; Cl, 14.37%. ^{1}H NMR δ_H (in ppm, $CDCl_3$, 400 MHz) 5.28-5.45 (3H, mm, C*H*=C*H*, C*H*OC(O)); 4.42 (2H, dd, *J*=3.8, 3.8 Hz, RC(O)OC$H_a$$H_b$CHCH$_2$OC(O)R'); 4.25 (2H, dd, *J*=7.0, 6.9 Hz, RC(O)OCH$_2$CHC$H_a$$H_b$OC(O)R'); 2.32 (4H, t, *J*=7.4 Hz, 2-$CH_{2,\ Palm}$, 2-CH_2); 2.06-1.94 (4H, m, 8-CH_2, 11-CH_2); 1.65-1.55 (4H, m, 3-$CH_{2,\ Palm}$, 3-CH_2); 1.20-1.38 (44H, m, 4-15-$CH_{2,\ Palm}$, 4-7-CH_2, 12-17-CH_2); 0.87 (6H, t,

J=6.6 Hz, 16-C*H*$_3$, 18-C*H*$_3$). ^{13}C NMR δ_C (in ppm, $CDCl_3$, 100 MHz) 173.27, 173.30 (C1); 129.93, 130.24 (C9, C10); 34.16 (C2); 32.14 (C14); 32.12 (C16); 29.28-29.99 (C4-C13, C4-C7, C12-C15); 27.38, 27.44 (C11, C8); 25.01 (C3); 22.91 (C17); 14.33 (C16, C18): oleoyl and palmitoyl fragments; 161.54 (C1): trichloroacetyl fragment; 74.80 (C2); 61.64 (C3, C1): glycerol fragment.

1,2-Diacetyl-3-oleoyl-*sn*-glycerol 35. Obtained from 1-*O*-*tert*-butyldimethylsilyl-2-acetyl-3-oleoyl-*sn*-glycerol (**20**; 0.513 g, 1.00 mmol) and acetic anhydride (0.284 mL, 3.00 mmol). Yield: 0.410 g (93%, colourless oil); R_f (pentane/toluene/EtOAc = 40:50:10, v/v/v) = 0.25; $[\alpha]_D^{20}$= +1.23 (*c* 10.83, $CHCl_3$); Found: C, 68.08; H, 10.12%. $C_{25}H_{44}O_6$ (440.61) requires C, 68.15; H, 10.06%. ^{1}H NMR δ_H (in ppm, $CDCl_3$, 400 MHz) 5.39-5.27 (2H, m, C*H*=C*H*); 5.25-5.22 (1H, m, C*H*OC(O)); 4.33-4.25 (2H, m, RC(O)OCH_aH$_b$CHCH$_2$OC(O)CH$_3$, RC(O)OCH$_2$CHCH$_a$$H_b$OC(O)CH$_3$); 4.14 (2H, m, RC(O)OCH$_a$$H_b$CHCH$_2$OC(O)CH$_3$, RC(O)OCH$_2CHCH_aH_b$OC(O)CH$_3$); 2.31 (2H, t, *J*=7.7 Hz, 2-C*H*$_2$); 2.07 (3H, s, 2-C*H*$_3$); 2.06 (3H, s, 2-C*H*$_3$); 2.05-1.95 (4H, m, 8-C*H*$_2$, 11-C*H*$_2$); 1.63-157 (2H, m, 3-C*H*$_2$); 1.21-1.39 (20H, m, 4-7-C*H*$_2$, 12-17-C*H*$_2$); 0.87 (3H, t, *J*= 7.0 Hz, 18-CH$_3$). ^{13}C NMR δ_C (in ppm, $CDCl_3$, 100 MHz) 173.49 (C1); 129.92, 130.23 (C9, C10); 34.23 (C2); 32.11 (C16); 29.27-29.97 (C4-C7, C12-C15); 27.37, 27.43 (C11, C8); 25.05 (C3); 22.88 (C17); 14.31 (C18): oleoyl fragment; 170.28, 170.69 (C1); 20.89, 21.09 (C2): both acetyl fragments; 69.34 (C2); 62.50 (C1); 62.20 (C3): glycerol fragment.

1-Oleoyl-2,3-diacetyl-*sn*-glycerol 36. Obtained from 1-oleoyl-2-acetyl-3-*O*-triisopropylsilyl-*sn*-glycerol (**21**; 0.555 g, 1.00 mmol) and acetic anhydride (0.284 mL, 3.00 mmol). Yield: 0.401 g (91%, colourless oil); $[\alpha]_D^{20}$= -1.29 (*c* 8.93, $CHCl_3$). All other physicochemical and spectral characteristics were identical with those of **35**.

1-Oleoyl-2-acetyl-3-palmitoyl-*sn*-glycerol 37. Synthesized from 1-oleoyl-2-acetyl-3-*O*-triisopropylsilyl-*sn*-glycerol (**21**; 0.555 g, 1.00 mmol) and palmitic anhydride (1.484 g, 3.00 mmol). Yield: 0.611 g (96%, colourless oil); R_f (pentane/toluene/EtOAc = 40:50:10, v/v/v) = 0.57; $[\alpha]_D^{20}$= 0.00 (*c* 5.11, $CHCl_3$); Found: C, 73.66; H, 11.20%.

$C_{39}H_{72}O_6$ (636.98) requires C, 73.54; H, 11.39%. ^{1}H NMR δ_H (in ppm, $CDCl_3$, 400 MHz) 5.30-5.38 (2H, m, C*H*=C*H*); 5.22-5.26 (1H, m, C*H*OC(O)); 4.30 (2H, dd, *J*=4.4, 4.2 Hz, RC(O)OCH_aH_bCHCH_2OC(O)R'); 4.14 (2H, dd, *J*=5.9, 5.9 Hz, RC(O)OCH_2CHCH_aH_bOC(O)R'); 2.31 (4H, t, *J*=7.5 Hz, 2-C$H_{2,\ Palm}$, 2-CH_2); 2.07 (3H, s, 2-CH_3); 2.04-1.96 (4H, m, 8-CH_2, 11-CH_2); 1.63-1.57 (4H, m, 3-C$H_{2,\ Palm}$, 3-CH_2); 1.20-1.40 (44H, m, 4-15-C$H_{2,\ Palm}$, 4-7-CH_2, 12-17-CH_2); 0.87 (6H, t, *J*=7.1 Hz, 16-CH_3, 18-CH_3). ^{13}C NMR δ_C (in ppm, $CDCl_3$, 100 MHz) 173.50, 173.53 (C1); 129.93, 130.23 (C9, C10); 34.25 (C2); 32.14 (C14); 32.12 (C16); 29.29-29.98 (C4-C13, C4-C7, C12-C15); 27.38, 27.43 (C11, C8); 25.07 (C3); 22.91 (C17); 14.33 (C16, C18): oleoyl and palmitoyl fragments; 170.28 (C1); 21.09 (C2): acetyl fragment; 69.40 (C2); 62.23 (C3, C1): glycerol fragment.

1,2-Dioleoyl-3-acetyl-*sn*-glycerol 38. Acquired from 1,2-dioleoyl-3-*O*-triisopropylsilyl-*sn*-glycerol (**23**; 0.777 g, 1.00 mmol) and acetic anhydride (0.284 mL, 3.00 mmol). Yield: 0.603 g (91%, colourless oil); R_f (pentane/toluene/EtOAc = 40:50:10, v/v/v) = 0.53; $[\alpha]_D^{20}$= -0.68 (*c* 7.60, $CHCl_3$); Found: C, 74.22; H, 11.28%. $C_{41}H_{74}O_6$ (663.02) requires C, 74.27; H, 11.25%. ^{1}H NMR δ_H (in ppm, $CDCl_3$, 400 MHz) 5.37-5.31 (4H, m, C*H*=C*H*); 5.28-5.24 (1H, m, C*H*OC(O)); 4.24-4.33 (2H, m, RC(O)OCH_2CHCH_aH_bOC(O)CH_3); 4.14 (2H, dd, *J*= 5.9, 6.0 Hz, RC(O)OCH_aH_bCHCH_2OC(O)CH_3); 2.25-2.39 (4H, m, 2-CH_2); 2.06 (3H, s, 2-CH_3); 2.01 (8H, m, 8-CH_2, 11-CH_2); 1.65-1.57 (4H, m, 3-CH_2); 1.20-140 (40H, m, 4-7-CH_2, 12-17-CH_2); 0.88 (6H, t, *J*= 7.1 Hz, 18-CH_3). ^{13}C NMR δ_C (in ppm, $CDCl_3$, 100 MHz) 173.09, 173.47 (C1); 129.91, 129.92, 130.24, 130.25 (C9, C10); 34.25, 34.41 (C2); 32.12 (C16); 29.25-29.98 (C4-C7, C12-C15); 27.39, 27.44 (C11, C8); 25.06, 25.10 (C3); 22.90 (C17); 14.33 (C18): both oleoyl fragments; 170.68 (C1); 20.90 (C2): acetyl fragment; 69.03 (C2); 62.58 (C3); 62.28 (C1): glycerol fragment.

1,2,3-Trioleoyl glycerol 39. Obtained from 1,2-dioleoyl-3-*O*-triisopropylsilyl-*sn*-glycerol (**23**; 0.777 g, 1.00 mmol) and oleic anhydride (1.641 g, 3.00 mmol). Yield: 0.823 g (93%, colourless oil); R_f (pentane/EtOAc = 90:10, v/v) = 0.72; Found: C, 77.37; H, 11.81%. $C_{57}H_{104}O_6$ (885.43) requires C, 77.32; H, 11.84%. ^{1}H NMR δ_H (in ppm, $CDCl_3$, 400 MHz) 5.38-5.30 (6H, m, C*H*=C*H*); 5.26 (1H, m, C*H*OC(O)); 4.29 (2H, dd, *J*= 4.4, 4.4 Hz,

RC(O)OCH$_2$CHCH_aH_bOC(O)R); 4.14 (2H, dd, *J*= 6.2, 5.9 Hz, RC(O)OCH_aH_bCHCH$_2$OC(O)R); 2.25-2.39 (6H, m, 2-CH_2); 2.01 (12H, m, 8-CH_2, 11-CH_2); 1.65-1.57 (6H, m, 3-CH_2); 1.20-1.40 (60H, m, 4-7-CH$_2$, 12-17-CH_2); 0.87 (9H, t, *J*= 7.0 Hz, 18-CH_3). ^{13}C NMR δ_C (in ppm, CDCl$_3$, 100 MHz) 172.84, 173.29 (C1); 129.71, 129.74, 130.05, 130.07 (C9, C10); 34.07, 34.23 (C2); 31.96 (C16); 29.24-29.98 (C4-C7, C12-C15); 27.21, 27.26 (C11, C8); 24.89, 24.93 (C3); 22.73 (C17); 14.15 (C18): oleoyl fragments; 68.93 (C2); 62.14 (C1, C3): glycerol fragment.

1-Acetyl-2-oleoyl-3-palmitoyl-*sn*-glycerol 40. Obtained from 1-*O*-triisopropylsilyl-2-oleoyl-3-palmitoyl-*sn*-glycerol (**24**; 0.751 g, 1.00 mmol) and acetic anhydride (0.284 mL, 3.00 mmol). Yield: 0.586 g (92%, colourless oil); R_f (pentane/toluene/EtOAc = 40:50:10, v/v/v) = 0.52; $[\alpha]_D^{20}$= +0.72 (*c* 11.73, CHCl$_3$); Found: C, 73.57; H, 11.40%. $C_{39}H_{72}O_6$ (636.98) requires C, 73.54; H, 11.39%. ^{1}H NMR δ_H (in ppm, CDCl$_3$, 400 MHz) 5.38-5.30 (2H, m, C*H*=C*H*); 5.28-5.24 (1H, m, C*H*OC(O)); 4.24-4.33 (2H, m, CH$_3$C(O)OCH_aH_bCHCH$_2$OC(O)R); 4.14 (2H, dd, *J*=6.0, 6.0 Hz, CH$_3$C(O)OCH$_2$CHCH_aH_bOC(O)R); 2.35-2.26 (4H, m, 2-C$H_{2,\ Palm}$, 2-CH_2); 2.06 (3H, s, 2-CH_3); 2.04-1.96 (4H, m, 8-CH_2, 11-CH_2); 1.65-1.55 (4H, m, 3-C$H_{2,\ Palm}$, 3-CH_2); 1.20-1.40 (44H, m, 4-15-C$H_{2,\ Palm}$, 4-7-CH_2, 12-17-CH_2); 0.87 (6H, t, *J*=7.1 Hz, 16-CH_3, 18-CH_3). ^{13}C NMR δ_C (in ppm, CDCl$_3$, 100 MHz) 173.09, 173.50 (C1); 129.90, 130.24 (C9, C10); 34.26, 34.41 (C2); 32.14 (C16, C14); 29.25-29.98 (C4-C13, C4-C7, C12-C15); 27.38, 27.44 (C11, C8); 25.08 (C3); 22.91 (C17); 14.33 (C16, C18): oleoyl and palmitoyl fragments; 170.68 (C1); 20.90 (C2): acetyl fragment; 69.03 (C2); 62.58 (C1); 62.27 (C3): glycerol fragment.

1,3-Dipalmitoyl-2-oleoyl glycerol 41. Obtained from 1-*O*-triisopropylsilyl-2-oleoyl-3-palmitoyl-*sn*-glycerol (**24**; 0.751 g, 1.00 mmol) and palmitic anhydride (1.484 g, 3.00 mmol). Yield: 0.775 g (93%); white solid: m.p. 37.4-38.0 ^{o}C; R_f (pentane/toluene/EtOAc = 40:50:10, v/v/v) = 0.56; lit.[30] m.p 35.0-37.5 ^{o}C; Found: C, 76.43; H, 12.04%. $C_{53}H_{100}O_6$ (833.36) requires C, 76.39; H, 12.09%. ^{1}H NMR δ_H (in ppm, CDCl$_3$, 400 MHz) 5.38-5.30 (2H, m, C*H*=C*H*); 5.28-5.24 (1H, m, C*H*OC(O)); 4.29 (2H, dd, *J*=4.4, 4.4 Hz, RC(O)OCH_aH_bCHCH$_2$OC(O)R); 4.14 (2H, dd, *J*=6.0, 5.8 Hz, RC(O)OCH$_2$CHCH_aH_bOC(O)R); 2.34-2.26 (6H, m, 2-C$H_{2,\ Palm}$, 2-CH_2); 2.00 (4H, m, 8-CH_2, 11-CH_2); 1.65-1.55 (6H, m, 3-C$H_{2,\ Palm}$, 3-CH_2); 1.20-1.40

(68H, m, 4-15-C$H_{2,\ Palm}$, 4-7-CH_2, 12-17-CH_2); 0.88 (9H, t, *J*=6.9 Hz, 16-CH_3, 18-CH_3). ^{13}C NMR δ_C (in ppm, $CDCl_3$, 100 MHz) 173.51 (C1); 129.91, 130.24 (C9, C10); 34.28, 34.42 (C2); 32.15 (C16, C14); 29.28-29.99 (C4-C13, C4-C7, C12-C15); 27.40, 27.45 (C11, C8); 25.09 (C3); 22.91 (C17); 14.34 (C16, C18): oleoyl and palmitoyl fragments; 69.10 (C2); 62.31 (C1, C3): glycerol fragment.

1-Oleoyl-2-acetyl-*sn*-glycerol 42. Acquired from 1-oleoyl-2-acetyl-3-trichloroacetyl-*sn*-glycerol (**28**; 0.544 g, 1.00 mmol). Yield: 0.398 g (100%, colourless oil); R_f (toluene/EtOAc = 80:20, v/v) = 0.24; $[\alpha]_D^{20}$= -5.42 (*c* 5.07, $CHCl_3$); Found: C, 70.01; H, 10.60%. $C_{23}H_{42}O_5$ (398.58) requires C, 69.31; H, 10.62%. ^{1}H NMR δ_H (in ppm, $CDCl_3$, 400 MHz) 5.37-5.29 (2H, m, C*H*=C*H*); 5.06 (1H, tt, *J*=4.9, 4.9 Hz, C*H*OC(O)); 4.32 (1H, dd, *J*=4.4, 4.4 Hz, RC(O)OCH_aH$_b$CHCH$_2$OH); 4.22 (1H, dd, *J*=5.5, 5.7 Hz, RC(O)OCH$_a$$H_b$CHCH$_2$OH); 3.75-3.68 (2H, m, RC(O)OCH$_2$CHC$H_a$$H_b$OH); 2.32 (2H, t, *J*=7.5 Hz, 2-CH_2); 2.10 (3H, s, 2-CH_3); 2.03-1.97 (4H, m, 8-CH_2, 11-CH_2); 1.65-1,57 (2H, m, 3-CH_2); 1.20-1.40 (20H, m, 4-7-CH_2, 12-17-CH_2); 0.87 (3H, t, *J*= 6.6 Hz, 18-CH_3). ^{13}C NMR δ_C (in ppm, $CDCl_3$, 100 MHz) 174.02 (C1); 129.92, 130.25 (C9, C10); 34.29 (C2); 32.11 (C16); 29.29-29.98 (C4-C7, C12-C15); 27.37, 27.43 (C11, C8); 25.08 (C3); 22.89 (C17); 14.32 (C18): oleoyl fragment; 170.78 (C1); 21.21 (C2): acetyl fragment; 72.58 (C2); 62.17 (C1); 61.63 (C3): glycerol fragment.

2-Acetyl-3-oleoyl-*sn*-glycerol 43. Obtained from 1-trichloroacetyl-2-acetyl-3-oleoyl-*sn*-glycerol (**29**; 0.544 g, 1.00 mmol). Yield: 0.399 g (100%, colourless oil); $[\alpha]_D^{20}$= +5.48 (*c* 3.25, $CHCl_3$). All other physicochemical and spectral characteristics were identical with those of **42**.

1,**2-Dioleoyl-*sn*-glycerol 44**. Produced from 1,2-dioleoyl-3-trichloroacetyl-*sn*-glycerol (**30**; 0.766 g, 1.00 mmol). Yield: 0.621 g (100%, colourless oil); R_f (toluene/EtOAc = 80:20, v/v) = 0.50; $[\alpha]_D^{20}$= -2.85 (*c* 4.16, $CHCl_3$); lit.[94] $[\alpha]_D^{20}$= -2.5 (*c* 3.0, $CHCl_3$); Found: C, 75.50; H, 11.66%. $C_{39}H_{72}O_5$ (620.99) requires C, 75.43; H, 11.69%.^{1}H NMR δ_H (in ppm, $CDCl_3$, 400 MHz) 5.36-5.28 (4H, m, C*H*=C*H*); 5.07 (1H, tt, *J*=4.9, 4.9 Hz, C*H*OC(O)); 4.31 (1H, dd, *J*=4.7, 4.4 Hz, RC(O)OCH_aH$_b$CHCH$_2$OH); 4.22 (1H, dd, *J*=5.8, 5.5 Hz,

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RC(O)OCH$_a$*H$_b$*CHCH$_2$OH); 3.72 (2H, d, *J*=5.5 Hz, RC(O)OCH$_2$CHC*H$_a$H$_b$*OH); 2.32 (4H, overlapping tt, 2-C*H$_2$*); 2.07-1.93 (8H, m, 8-C*H$_2$*, 11-C*H$_2$*); 1.65-1.56 (4H, m, 3-C*H$_2$*); 1.20-1.40 (40H, m, 4-7-C*H$_2$*, 12-17-C*H$_2$*); 0.87 (6H, t, *J*= 6.9 Hz, 18-CH$_3$). ^{13}C NMR δ_C (in ppm, CDCl$_3$, 100 MHz) 173.61, 173.96 (C1); 129.91, 130.24 (C9, C10); 34.30, 34.48 (C2); 32.12 (C16); 29.28-29.98 (C4-C7, C12-C15); 27.38, 27.44 (C11, C8); 25.09, 25.14 (C3); 22.90 (C17); 14.32 (C18): oleoyl fragments; 72.33 (C2); 62.23 (C1); 61.76 (C3): glycerol fragment.

1-*O*-Hexadecyl-2-acetyl-*sn*-glycerol 45. Synthesized from 1-*O*-hexadecyl-2-acetyl-3-trichloroacetyl-*sn*-glycerol (**31**; 0.504 g, 1.00 mmol). Yield: 0.359 g (100%); white solid, m.p. 35.5-36.0 ^{o}C (from pentane); R$_f$ (toluene/EtOAc = 80:20, v/v) = 0.23; $[\alpha]_D^{20}$= -5.44 (*c* 2.39, CHCl$_3$); lit.[95] $[\alpha]_D^{20}$= -11.1 (*c* 0.4, CHCl$_3$); Found: C, 70.51; H, 11.77%. C$_{21}$H$_{42}$O$_4$ (358.56) requires C, 70.34; H, 11.81%. ^{1}H NMR δ_H (in ppm, CDCl$_3$, 400 MHz) 4.98 (1H, tt, *J*=4.8, 4.8 C*H*OC(O)); 3.83-3.78 (2H, m, ROCH$_2$CHC*H$_a$H$_b$*OH); 3.65-2.59 (2H, m, HOCH$_2$CHC*H$_a$H$_b$*OR); 3.49-3.41 (2H, m, 1-C*H$_2$*); 2.10 (3H, s, 2-C*H$_3$*); 1.55 (2H, m, 2-C*H$_2$*); 1.15-1.35 (26H, m, 3-15-C*H$_2$*); 0.87 (3H, t, *J*= 7.0 Hz, 16-C*H$_3$*). ^{13}C NMR δ_C (in ppm, CDCl$_3$, 100 MHz) 72.15 (C1); 32.14 (C2); 29.57-29.91 (C4-C14); 26.24 (C3); 22.90 (C15); 14.33 (C16): hexadecyl fragment; 171.08 (C1); 21.35 (C2): acetyl fragment; 73.29 (C2); 70.19 (C1); 63.15 (C3): glycerol fragment.

2-Acetyl-3-*O*-hexadecyl-*sn*-glycerol 46. Obtained from 1-trichloroacetyl-2-acetyl-3-*O*-hexadecyl-*sn*-glycerol (**32**; 0.504 g, 1.00 mmol). Yield: 0.359 g (100%, white solid); $[\alpha]_D^{20}$= +5.98 (*c* 2.08, CHCl$_3$). All other physicochemical and spectral characteristics were identical with those of **45**.

1-Oleoyl-3-acetyl-*sn*-glycerol 47. Obtained from 1-oleoyl-2-trichloroacetyl-3-acetyl-*sn*-glycerol (**33**; 0.544 g, 1.00 mmol). Yield: 0.398 g (100%, colourless oil); R$_f$ (toluene/EtOAc = 80:20, v/v) = 0.29; $[\alpha]_D^{20}$= -0.28 (*c* 9.15, CHCl$_3$); Found: C, 69.19; H, 10.70%. C$_{23}$H$_{42}$O$_5$ (398.58) requires C, 69.31; H, 10.62%. ^{1}H NMR δ_H (in ppm, CDCl$_3$, 400 MHz) 5.37-5.29 (2H, m, C*H*=C*H*); 4.02-4.23 (5H, m, RC(O)OC*H$_a$H$_b$*CHC*H$_a$H$_b$*O(O)CCH$_3$, C*H*OH); 2.34 (2H, t, *J*=7.7 Hz, 2-C*H$_2$*); 2.09 (3H, s, 2-C*H$_3$*); 2.04-1.96 (4H, m, 8-C*H$_2$*, 11-C*H$_2$*); 1.66-1.56 (2H, m, 3-C*H$_2$*); 1.20-

140 (20H, m, 4-7-CH_2, 12-17-CH_2); 0.87 (3H, t, *J*=7.0 Hz, 18-CH_3). ^{13}C NMR δ_C (in ppm, $CDCl_3$, 100 MHz) 174.12 (C1); 129.93, 130.24 (C9, C10); 34.29 (C2); 32.11 (C16); 29.29-29.97 (C4-C7, C12-C15); 27.37, 27.43 (C11, C8); 25.08 (C3); 22.88 (C17); 14.32 (C18): oleoyl fragment; 171.26 (C1); 20.99 (C2): acetyl fragment; 68.49 (C2); 65.46 (C3); 65.23 (C1): glycerol fragment.

1-Oleoyl-3-palmitoyl-*sn*-glycerol 48. Produced from 1-oleoyl-2-trichloroacetyl-3-palmitoyl-*sn*-glycerol (**34**; 0.740 g, 1.00 mmol). Yield: 0.595 g (100%); white solid, m.p. 45.5-47.0 ^{o}C (from pentane); lit.[84] m.p. 45-46^{o}C; R_f (toluene/EtOAc = 80:20, v/v) = 0.52; Found: C, 74.81; H, 11.80%. $C_{37}H_{70}O_5$ (594.95) requires C, 74.69; H, 11.86%. ^{1}H NMR δ_H (in ppm, $CDCl_3$, 400 MHz) 5.37-5.31 (2H, m, C*H*=C*H*); 4.02-4.28 (5H, m, RC(O)OCH_aH_bCHCH_aH_bOC(O)R', C*H*OH); 2.34 (4H, t, *J*=7.7 Hz, 2-C$H_{2,\ Palm}$, 2-CH_2); 2.04-1.96 (4H, m, 8-CH_2, 11-CH_2); 1.66-1.58 (4H, m, 3-C$H_{2,\ Palm}$, 3-CH_2); 1.20-1.40 (44H, m, 4-15-C$H_{2,\ Palm}$, 4-7-CH_2, 12-17-CH_2); 0.87 (6H, t, *J*=6.9 Hz, 16-CH_3, 18-CH_3). ^{13}C NMR δ_C (in ppm, $CDCl_3$, 100 MHz) 174.10, 174.13 (C1); 129.93, 130.24 (C9, C10); 34.32 (C2); 32.14 (C14); 32.12 (C16); 29.31-29.98 (C4-C13, C4-C7, C12-C15); 27.38, 27.44 (C11, C8); 25.11 (C3); 22.91 (C17); 14.33 (C16, C18): oleoyl and palmitoyl fragments; 68.61 (C2); 65.26 (C3, C1): glycerol fragment.

1-Oleoyl-2-[*R*-(-)-α-methoxy-α-trifluoromethylphenylacetyl]-3-acetyl-*sn*-glycerol, 49. The compound was prepared from 1-oleoyl-3-acetyl-*sn*-glycerol (**47**; 0.399 g, 1.00 mmol) and *R*-(-)-α-methoxy-α-trifluoromethylphenylacetyl chloride (0.224 mL, 1.20 mmol) at room temperature for 18 h according to the above general procedure with an exception that after removing the solvents, the residue was taken in toluene/ethyl acetate (98:2, v/v, 5 mL) and the solution was passed through a silica gel pad (~5 g) prepared in the same solvent system. The support was washed with this eluant (50 mL), fractions containing the product were combined and the solution was concentrated under reduced pressure to afford the crude Mosher ester **49**, which was examined next by ^{1}H and ^{13}C NMR without further purification. Yield Calcd for $C_{33}H_{49}F_3O_7$ (614.73): 0.553 g (90%, colorless oil); R_f (pentane/toluene/EtOAc = 40:50:10, v/v/v) = 0.45. ^{1}H NMR δ_H (in ppm, $CDCl_3$, 400 MHz) 7.50-7.60 (2H, m, *Ar*-ring); 7.33-7.45 (3H, m, *Ar*-ring); 5.55-5.49 (1H, m,

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C*H*OC(O)); 5.37-5.31 (2H, m, C*H*=C*H*); 4.41 (1H, dd, *J*=3.7, 3.7 Hz, $ROCH_2CHCH_aH_bO(O)CCH_3$); 4.34 (1H, dd, *J*=3.8, 3.8 Hz, $ROCH_2CHCH_aH_bO(O)CCH_3$); 4.18 (1H, dd, *J*=7.1, 7.1 Hz, $RC(O)OCH_aH_bCHCH_2O(O)CCH_3$); 4.12 (1H, dd, *J*=6.4, 6.4 Hz, $RC(O)OCH_aH_bCHCH_2O(O)CCH_3$); 3.56 (3H, s, C*H*$_3$CO); 2.29 (2H, t, *J*=7.1 Hz, 2-C*H*$_2$); 1.98 (3H, s, 2-C*H*$_3$); 2.05-1.96 (4H, m, 8-C*H*$_2$, 11-C*H*$_2$); 1.59-1.53 (2H, m, 3-C*H*$_2$); 1.20-1.40 (20H, m, 4-7-C*H*$_2$, 12-17-C*H*$_2$); 0.87 (3H, t, *J*=7.0 Hz, 18-CH_3). ^{13}C NMR δ_C (in ppm, $CDCl_3$, 100 MHz) 173.26 (C1); 130.24, 129.94 (C9, C10); 34.13 (C2); 32.12 (C16); 29.27-29.98 (C4-C7, C12-C15); 27.38, 27.44 (C11, C8); 24.93 (C3); 22.89 (C17); 14.32 (C18): oleoyl fragment; 170.44 (C1); 20.68 (C2): acetyl fragment; 166.15 (-*C*(O)-); 127.53, 128.63, 132.19 (C1-C6, Ar-ring); 123.50 (q, *J*=289.2 Hz, F_3C-); 55.62 (*C*H_3O-): MTPA-fragment; 71.57 (C2); 62.07 (C1); 62.09 (C3): glycerol fragment.

1-Oleoyl-2,3-diacetyl-*sn*-glycerol 50. Obtained from 1-oleoyl-2-acetyl-3-trichloroacetyl-*sn*-glycerol (**28**; 0.544 g, 1.00 mmol) via **42** and acetyl chloride (0.142 mL; 2.00 mmol). Overall yield: 0.418 g (95%, colourless oil); $[\alpha]_D^{20}$= -1.25 (*c* 9.33, $CHCl_3$). All other physicochemical and spectral characteristics were identical with those of **35** and **36**.

1-Palmitoyl-2-acetyl-3-oleoyl-*sn*-glycerol 51. Synthesized from 1-trichloroacetyl-2-acetyl-3-oleoyl-*sn*-glycerol (**29**; 0.544 g; 1.00 mmol) via **43** and palmitoyl chloride (0.606 mL; 2.00 mmol). Overall yield: 0.598 g (94%, colourless oil); $[\alpha]_D^{20}$= 0.00 (*c* 8.69, $CHCl_3$). All other physicochemical and spectral characteristics were identical with those of **37**.

1-Oleoyl-2-palmitoyl-3-acetyl-*sn*-glycerol 52. Acquired from 1-oleoyl-2-trichloroacetyl-3-acetyl-*sn*-glycerol (**33**; 0.544 g, 1.00 mmol) via **47** and palmitoyl chloride (0.606 mL; 2.00 mmol). Yield: 0.580 g (91%, colourless oil); R_f (pentane/toluene/EtOAc = 40:50:10, v/v/v) = 0.55; $[\alpha]_D^{20}$= -0.64 (*c* 8.15, $CHCl_3$); Found: C, 73.50; H, 11.31%. $C_{39}H_{72}O_6$ (636.98) requires C, 73.54; H, 11.39%.^{1}H NMR δ_H (in ppm, $CDCl_3$, 400 MHz) 5.37-5.31 (2H, m, C*H*=C*H*); 5.29-5.23 (1H, m, C*H*OC(O)); 4.30-4.26 (2H, m, $RC(O)OCH_aH_bCHCH_2O(O)CCH_3$); 4.14 (2H, dd,

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J=6.0, 6.2 Hz, RC(O)OCH$_2$CHCH_aH_bO(O)CCH$_3$); 2.35-2.26 (4H, m, 2-C$H_{2,\ Palm}$, 2-CH_2); 2.06 (3H, s, 2-CH_3); 2.04-1.96 (4H, m, 8-CH_2, 11-CH_2); 1.65-1.55 (4H, m, 3-C$H_{2,\ Palm}$, 3-CH_2); 1.20-1.40 (44H, m, 4-15-C$H_{2,\ Palm}$, 4-7-CH_2, 12-17-CH_2); 0.87 (6H, t, *J*=7.1 Hz, 16-CH_3, 18-CH_3). ^{13}C NMR δ_C (in ppm, CDCl$_3$, 100 MHz) 173.17, 173.47 (C1); 129.92, 130.23 (C9, C10); 34.25, 34.42 (C2); 32.14 (C14, C16); 29.28-29.98 (C4-C13, C4-C7, C12-C15); 27.38, 27.44 (C11, C8); 25.06, 25.11 (C3); 22.90 (C17); 14.32 (C16, C18): oleoyl and palmitoyl fragments; 170.68 (C1); 20.90 (C2): acetyl fragment; 69.01 (C2); 62.58 (C3); 62.28 (C1): glycerol fragment.

1-Oleoyl-2,3-palmitoyl-*sn*-glycerol 53. Obtained from 1-oleoyl-2-trichloroacetyl-3-palmitoyl-*sn*-glycerol (**34**; 0.740 g; 1.00 mmol) via **48** and palmitoyl chloride (0.606 mL; 2.00 mmol). Yield: 0.758 g (91%); white solid, m.p. 34.0-35.0 ^{o}C; R_f (pentane/toluene/EtOAc = 40:50:10, v/v/v) = 0.54; $[\alpha]_D^{20}$= 0.00 (c 7.05, CHCl$_3$); lit.[30] m.p 29.8-34.5 ^{o}C; Found: C, 76.40; H, 12.12%. $C_{53}H_{100}O_6$ (833.36) requires C, 76.39; H, 12.09%; ^{1}H NMR δ_H (in ppm, CDCl$_3$, 400 MHz) 5.37-5.31 (2H, m, C*H*=C*H*); 5.28-5.24 (1H, m, C*H*OC(O)); 4.29 (2H, dd, *J*=4.4, 4.4 Hz, RC(O)OCH_aH_bCHCH$_2$OC(O)R'); 4.14 (2H, dd, *J*=6.2, 5.9 Hz, RC(O)OCH$_2$CHCH_aH_bOC(O)R'); 2.35-2.25 (6H, m, 2-C$H_{2,\ Palm}$, 2-CH_2); 2.05-1.96 (4H, m, 8-CH_2, 11-CH_2); 1.65-1.56 (6H, m, 3-C$H_{2,\ Palm}$, 3-CH_2); 1.20-1.40 (68H, m, 4-15-C$H_{2,\ Palm}$, 4-7-CH_2, 12-17-CH_2); 0.88 (9H, t, *J*=7.3 Hz, 16-CH_3, 18-CH_3). ^{13}C NMR δ_C (in ppm, CDCl$_3$, 100 MHz) 173.08, 173.47, 173.50 (C1); 129.92, 130.23 (C9, C10); 34.25, 34.27, 34.44 (C2); 32.15 (C16, C14); 29.31-29.98 (C4-C13, C4-C7, C12-C15); 27.39, 27.44 (C11, C8); 25.08, 25.13 (C3); 22.91 (C17); 14.32 (C16, C18): oleoyl and palmitoyl fragments; 69.09 (C2); 62.32 (C1, C3): glycerol fragment.