Dichlorination of olefins with NCS/Ph$_3$P

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General. Melting points are uncorrected. All reagents were used as received from commercial suppliers unless otherwise noted. $^1$H NMR spectra (400 or 300 MHz) and $^{13}$C NMR spectra (125 or 100 or 75 MHz) were measured in the specified solvents. Chemical shifts are reported in ppm relative to the internal solvent signal [chloroform- $d$: 7.26 ppm ($^1$H NMR), 77.0 ppm ($^{13}$C NMR)]. FT-IR spectra were recorded for samples loaded as neat films on NaCl plates. Mass spectra were obtained according to the specified technique. Analytical thin layer chromatography (TLC) was performed using Kieselgel 60 F$_{254}$. Compounds were visualized with UV light and stained with anisaldehyde solution or phosphomolybdic acid solution.

Dichlorination of olefins with a 2:1 mixture of NCS/Ph$_3$P (Table 1):

Table 1, entry 1

$^[b]^{[b]}$tert-Butyldimethyl(2,2,11,12-tetrachlorododecyl)oxy)silane (10), $^[12]$-(($^[b]^[b]$tert-Butyldimethylsilyloxy)-2,11,11-trichlorododecan-1-ol (S1), and $^[12]$-(($^[b]^[b]$tert-Butyldimethylsilyloxy)-1,11,11-trichlorododecan-2-ol (S2)

![Chemical structure of compounds]

To a solution of olefin 4 (70.7 mg, 0.20 mmol) in CH$_2$Cl$_2$ (2 mL) were added Ph$_3$P (78.7 mg, 0.30 mmol) and NCS (80.1 mg, 0.60 mmol). After being stirred for 1 h at room temperature, the mixture was treated with sat. NaHCO$_3$ and poured into a separatory funnel where it was extracted with CH$_2$Cl$_2$. The phases were separated and the organic phase was washed with brine, dried over MgSO$_4$, filtered, and concentrated. The residue was purified by flash silica gel column chromatography ($n$-hexane to EtOAc/$n$-hexane 1:3) to yield tetrachloride 10 (76.5 mg, 89%) as a colorless oil. Further elution gave separable regioisomeric chlorohydrins S1 (3.2 mg, 4%) and S2 (2.4 mg, 3%), both as colorless oils. Tetrachloride 10: IR (neat) $\nu$ 2930, 2857, 1119, 839, 779 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 4.04 (m, 1H), 3.92 (s, 2H), 3.76 (dd, 1H, $J = 11.4, 5.0$ Hz), 3.65 (dd, 1H, $J = 11.4, 7.3$ Hz), 2.21-2.14 (m, 2H), 1.99 (m, 1H), 1.72 (m, 1H), 1.64-1.50 (m, 3H), 1.48-1.20 (m, 9H) 0.91 (s, 9H), 0.11 (s, 6H); $^{13}$CNMR (100 MHz, CDCl$_3$) $\delta$ 93.4, 72.1, 61.2, 48.2, 43.4, 35.0, 29.22, 29.20, 28.97, 28.89, 25.8, 25.7 (3C), 24.7, 18.2, -5.4 (2C);
MS m/z 437 (M+H)+, 73 (100%); HRMS (FAB) calcd for C_{18}H_{37}O_{35}Cl_{4}Si (MH+) 437.1368, found: 437.1328.

Chlorohydrin S1: IR (neat) ν 3404, 2928, 2857, 1119, 839, 779 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 4.04 (m, 1H), 3.92 (s, 2H), 3.79 (m, 1H), 3.66 (m, 1H), 2.22-2.13 (m, 2H), 1.80-1.64 (m, 2H), 1.64-1.50 (m, 3H), 1.45-1.28 (m, 9H), 0.91 (s, 9H), 0.11 (s, 6H); ¹³CNMR (75 MHz, CDCl₃) δ 93.5, 72.1, 67.0, 65.4, 43.5, 34.2, 29.23, 29.19, 29.02, 28.97, 26.3, 25.7 (3C), 24.7, 18.2, -5.4 (2C); MS m/z: 421 (M+H)+, 73 (100%); HRMS (FAB) calcd for C_{18}H_{37}O_{37}Cl_{35}ClSi (MH+) 421.1677, found 421.1681.

Table 1, entry 2

(1R*,2R*)-1,2-Dichlorocyclooctane (11)

The title compound was prepared according to the general procedure described for entry 1 using olefin 5 (26.0 µL, 0.20 mmol), Ph₃P (78.7 mg, 0.30 mmol), and NCS (80.1 mg, 0.60 mmol). (Reaction time: 1 h) Purification by flash silica gel column chromatography (n-hexane) gave dichloride 11 (33.8 mg, 93%) as a colorless oil. The ¹H NMR spectrum of compound 11 is in good agreement with that reported. Dichloride 11: IR (neat) ν 2926, 2857, 1462, 733 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 4.32-4.24 (m, 2H), 2.34-2.22 (m, 2H), 2.10-1.96 (m, 2H), 1.92-1.80 (m, 2H), 1.77-1.64 (m, 2H), 1.63-1.50 (m, 2H), 1.48-1.35 (m, 2H); ¹³CNMR (100 MHz, CDCl₃) δ 68.4, 33.6, 25.7, 25.4.
Table 1, entry 3

\((4S^*,5R^*,E)\)-Ethyl 4,5-dichlorohex-2-enoate (12) and \((E)\)-Ethyl 2,5-dichlorohex-3-enoate (13)

\[
\begin{array}{c}
\text{OEt} \\
\text{Cl} \\
\text{Cl} \\
\text{OEt}
\end{array}
\]

The title compounds were prepared according to the general procedure described for entry 1 using olefin 6 (28.0 mg, 0.20 mmol), \(\text{Ph}_3\text{P}\) (78.7 mg, 0.30 mmol), and NCS (80.1 mg, 0.60 mmol). (Reaction time: 5 min) Purification by flash silica gel column chromatography (toluene/\(n\)-hexane 1:3) gave dichloride 13 (8.9 mg, 21%) as a pale yellow oil. Further elution gave regioisomeric dichloride 12 (32.0 mg, 76%) as a pale yellow oil. The \(^1\)H NMR spectra of these compounds are in good agreement with those reported.\(^2\) **Dichloride 12:** IR (neat) \(\nu\) 2984, 2930, 1724, 1661, 1370, 1317, 1271, 1227, 1177, 1040, 974, 656 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 6.91 (dd, 1H, \(J = 15.6, 8.2\) Hz), 6.10 (d, 1H, \(J = 15.6\) Hz), 4.49 (dd, 1H, \(J = 8.2, 7.3\) Hz), 4.23 (q, 2H, \(J = 7.3\) Hz), 4.15 (dq, 1H, \(J = 7.3, 6.4\) Hz), 1.63 (d, 3H, \(J = 6.4\) Hz), 1.31 (t, 3H, \(J = 7.3\) Hz); \(^{13}\)CNMR (100 MHz, CDCl\(_3\)) \(\delta\) 165.3, 142.1, 125.3, 63.7, 60.9, 58.8, 21.9, 14.2. **Regioisomeric dichloride 13:** IR (neat) \(\nu\) 2928, 1744, 1443, 1371, 1267, 1177, 1018, 964, 677 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 6.00 (dd, 1H, \(J = 15.1, 6.9\) Hz), 5.93 (dd, 1H, \(J = 15.1, 7.3\) Hz), 4.77 (d, 1H, \(J = 7.3\) Hz), 4.55 (dq, 1H, \(J = 6.9, 6.4\) Hz), 4.26 (q, 2H, \(J = 6.9\) Hz), 1.62 (d, 3H, \(J = 6.9\) Hz), 1.32 (t, 3H, \(J = 6.9\) Hz) \(^{13}\)CNMR (100 MHz, CDCl\(_3\)) \(\delta\) 171.9, 137.5, 125.6, 62.5, 56.5, 55.8, 24.7, 14.0.

Table 1, entry 4

1,2-Dichloro-1,2-diphenylethane (14) and 2-Chloro-1,2-diphenylethanol (S3)

The title compounds were prepared according to the general procedure described for entry 1 using olefin 7 (36.1 mg, 0.20 mmol), \(\text{Ph}_3\text{P}\) (78.7 mg, 0.30 mmol), and NCS (80.1 mg, 0.60 mmol). (Reaction time: 15 min) Purification by flash silica gel column...
chromatography (toluene/n-hexane 1:3) gave dichloride 14 (38.5 mg, 77%) as a colorless amorphous solid. Further elution gave chlorohydrin S3 (7.6mg, 7%) as a colorless amorphous solid. The $^1$H NMR spectra of these compounds were in good agreement with those reported.\(^3\) **Dichloride 14** (1:1 diastereomeric mixture): IR (neat) $\nu$ 1454, 909, 733, 704, 675 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.47-7.36 (m, 5H), 7.24-7.13 (m, 5H), 5.25 (s, 1H), 5.23 (s, 1H); $^{13}$CNMR (100 MHz, CDCl$_3$) $\delta$ 138.3, 137.2, 129.0, 128.6, 128.5, 128.13, 128.08, 128.01, 67.7, 65.7. **Chlorohydrin S3** (1:1 diastereomeric mixture): IR (neat) $\nu$ 3420, 1454, 1053, 725, 696 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.30-6.99 (m, 10H), 5.02 (d, 0.5H, $J = 6.5$ Hz), 4.94 (d, 0.5H, $J = 6.5$ Hz), 4.93 (d, 0.5H, $J = 8.3$ Hz), 4.88 (d, 0.5H, $J = 8.3$ Hz), 2.98 (brs, 0.5H), 2.29 (brs, 0.5H); $^{13}$CNMR (100 MHz, CDCl$_3$) $\delta$ 139.4, 138.6, 137.6, 137.2, 128.7, 128.5, 128.43, 128.38, 128.34, 128.31, 128.2, 128.1, 127.9, 127.1, 126.9, 78.8, 78.2, 70.7, 66.9.

**Table 1, entry 5**

1,2-Dichloro-1,2-diphenylethane (14) and 2-Chloro-1,2-diphenylethanol (S3)

![Diagram](image)

The title compounds were prepared according to the general procedure described for entry 1 using olefin 8 (36.1 mg, 0.20 mmol), Ph$_3$P (78.7 mg, 0.30 mmol), and NCS (80.1 mg, 0.60 mmol). (Reaction time: 15 min) Purification by flash silica gel column chromatography (toluene/n-hexane 1:3) gave dichloride 14 (40.7 mg, 81%) as a colorless amorphous solid. Further elution gave chlorohydrin S3 (6.6 mg, 6%) as a colorless amorphous solid. The spectra of these products were identical with those obtained above.

**Table 1, entry 6**

(3$R^*$,4$R^*$)-3,4-Dichloro-6,8-bis(methoxymethoxy)-2,2-dimethyl-3,4-dihydro-$2H$-benzo[g]chromene-5,10-dione (15)
The title compound was prepared according to the general procedure described for entry 1 using olefin 9 (36.0 mg, 0.10 mmol), Ph₃P (39.3 mg, 0.15 mmol), and NCS (40.1 mg, 0.30 mmol). (Reaction time: 5 min) Purification by flash silica gel column chromatography (EtOAc/n-hexane 1:3) gave dichloride 15 (41.2 mg, 96%) as a yellow oil.

**Dichloride 15:** IR (neat) ν 2926, 1736, 1593, 1152 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.48 (d, 1H, J = 2.4 Hz), 7.16 (d, 1H, J = 2.4 Hz), 5.33, (s, 2H), 5.30 (d, 1H, J = 3.2 Hz), 5.28 (s, 2H), 4.37 (d, 1H, J = 3.2 Hz), 3.55 (s, 3H), 3.49 (s, 3H), 1.70 (s, 3H), 1.64 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 180.14, 178.96, 161.58, 159.30, 151.71, 134.36, 117.61, 115.22, 110.35, 108.08, 95.29, 94.19, 80.03, 63.61, 56.72, 56.58, 51.04, 26.33, 25.72; MS m/z: 430 (M⁺); HRMS (EI) calcd for C₁₉H₂₀O₇Cl₂ (M⁺): 430.0586, found: 430.0580.

**Dichlorination of epoxy cyclooctene 16 with a 2:1 or 1:1 mixture of NCS/Ph₃P (Scheme 3):** With a 1:1 combination of NCS/Ph₃P:

**(5R*,6S*,Z)-5,6-Dichlorocyclooct-1-ene (17)**

The title compound was prepared according to the general procedure described for Table 1 using epoxy olefin 16 (24.8 mg, 0.20 mmol), Ph₃P (157.3 mg, 0.60 mmol), and NCS (80.1 mg, 0.60 mmol). (Reaction time: 2.5 h) Purification by flash silica gel column chromatography (n-hexane) gave dichloride 17 (34.0 mg, 95%) as a colorless oil. The ¹H NMR spectrum of this compound was in good agreement with that reported.⁴ **Dichloride 17:** IR (neat) ν 2939, 750 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 5.80-5.65 (m, 2H), 4.65-4.45 (m, 2H), 2.67-2.63 (m, 2H), 2.36-2.27 (m, 2H), 2.11-1.97 (m, 4H); ¹³C NMR (100 MHz, CDCl₃) δ 128.47, 56.28, 27.80, 23.35.
With a 2:1 combination of NCS/Ph₃P:

\((1R^*,4S^*,5S^*,8S^*)-4,5\text{-Dichloro-9-oxabicyclo[6.1.0]nonane~(18)},~(1S^*,5S^*)-1,5\text{-Dichloro-9-oxabicyclo[3.3.1]nonane~(19)}\) and \((1R^*,2R^*,5S^*,6S^*)-2,5\text{-Dichloro-9-oxabicyclo[4.2.1]nonane~(20)}\)

The title compounds were prepared according to the general procedure described for Table 1 using epoxy olefin 16 (24.8 mg, 0.20 mmol), Ph₃P (78.7 mg, 0.30 mmol), and NCS (80.1 mg, 0.60 mmol). (Reaction time: 5 min) Purification by flash silica gel column chromatography (EtOAc/\(n\)-hexane 1:15) gave dichloride 18 (16.1 mg, 41%) as a colorless amorphous solid, a mixture of 19 (2.7 mg, 7%) and 20 (13.0 mg, 33%) as a colorless amorphous solid. The mixture of 19 and 20 was further subjected to flash column chromatography eluted with Et₂O/\(n\)-hexane (1:20) to afford sufficiently pure materials. The \(^{13}\)C NMR spectrum of compound 20 was identical to that reported.\(^5\)

**Dichloride 18:** IR (neat) \(\nu\) 2932, 1476, 1236, 1028, 934, 847, 789, 721, 621 \(\text{cm}^{-1}\); \(^1\)H NMR (400 MHz, CDCl₃) \(\delta\) 4.65-4.54 (m, 2H), 3.15 (m, 1H), 3.06 (m, 1H), 2.59-2.47 (m, 2H), 2.26-1.96 (m, 4H), 1.72-1.59 (m, 2H); \(^{13}\)C NMR (75 MHz, CDCl₃) \(\delta\) 61.4, 60.4, 55.9, 54.9, 30.5, 28.5, 24.0, 22.4. **Dichloride 19:** IR (neat) \(\nu\) 2926, 1738, 1485, 1123, 1044, 899, 870, 820 \(\text{cm}^{-1}\); \(^1\)H NMR (400 MHz, CDCl₃) \(\delta\) 4.33-4.27 (m, 2H), 3.93-3.90 (dd, 2H, \(J = 6.0, 5.5 \text{ Hz}\)), 2.35 (dt, 2H, \(J = 14.2, 3.7 \text{ Hz}\)), 2.22-2.12 (m, 4H), 2.07-1.94 (m, 2H); \(^{13}\)C NMR (75 MHz, CDCl₃) \(\delta\) 69.5, 57.6, 30.2, 24.0. **Dichloride 20:** IR (neat) \(\nu\) 2953, 2934, 1479, 1061, 932, 910, 793, 656 \(\text{cm}^{-1}\); \(^1\)H NMR (400 MHz, CDCl₃) \(\delta\) 4.62-4.51 (m, 2H), 4.29-4.19 (m, 2H), 2.40-2.29 (m, 2H), 2.18-1.96 (m, 6H); \(^{13}\)C NMR (75 MHz, CDCl₃) \(\delta\) 81.6, 60.4, 30.8, 26.2.

**Dichlorination of allylic alcohol 21 with NCS/Ph₃P in either 2:1 or 1:1 stoichiometry (Scheme 4):**

With a 1:1 combination of NCS/Ph₃P:
(E)-tert-Butyl((10-chlorodec-8-en-1-yl)oxy)diphenylsilane (22)

The title compound was prepared according to the general procedure described for Table 1 using alcohol 21 (82.1 mg, 0.20 mmol), Ph3P (78.7 mg, 0.30 mmol), and NCS (40.1 mg, 0.30 mmol). (Reaction time: 5 min) Purification by flash silica gel column chromatography (n-hexane) gave chloride 22 (80.2 mg, 94%) as a colorless oil.

Allylic chloride 22: IR (neat) ν 2930, 1111, 702 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.68-7.66 (m, 4H), 7.44-7.36 (m, 6H), 5.77 (dt, 1H, J = 14.8, 7.2 Hz), 5.61 (dt, 1H, 14.8, 6.8 Hz), 4.04 (d, 2H, 7.2 Hz), 3.66 (t, 2H, J = 6.4 Hz), 2.07-2.02 (m, 2H), 1.59-1.52 (m, 2H), 1.43-1.21 (m, 8H), 1.05 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 136.27, 135.56, 134.16, 129.47, 127.54, 125.82, 63.94, 45.55, 32.51, 32.02, 29.13, 29.05, 28.75, 26.86, 25.67, 19.21; MS m/z: 429 (M+H)⁺, 135 (100%); HRMS (FAB) calcd for C₂₆H₃₈OClSi⁺: 429.2380, found: 429.2366.

With a 2:1 combination of NCS/Ph₃P:

(E)-tert-Butyl((10-chlorodec-8-en-1-yl)oxy)diphenylsilane (22) and tert-Butyldiphenyl(((8R*,9S*)-8,9,10-trichlorodecyl)oxy)silane (23)

The title compounds were prepared according to the general procedure described for Table 1 using alcohol 21 (82.1 mg, 0.20 mmol), Ph₃P (78.7 mg, 0.30 mmol), and NCS (80.1 mg, 0.60 mmol). (Reaction time: 5 min) Purification by flash silica gel column chromatography (toluene/n-hexane 1:15) gave trichloride 23 (57.1 mg, 57%) and chloride 22 (20.9 mg, 24%), both as colorless oils.

Trichloride 23: IR (neat) ν 2931, 2857, 1111, 702 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.70-7.68 (m, 4H), 7.46-7.38 (m, 6H), 4.21-4.19 (m, 2H), 4.06 (dd, 1H, J = 12.4, 4.0 Hz), 3.94 (dd, 1H, J = 12.4, 3.2 Hz), 3.68 (t, 2H, J = 6.4 Hz), 2.05 (m, 1H), 1.81 (m, 1H), 1.58 (m, 2H), 1.47-1.28 (m, 8H), 1.07 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 135.55, 134.12, 129.48, 127.55, 63.88, 63.53, 62.06, 47.00,
34.06, 32.45, 29.07, 28.85, 26.87, 25.62, 25.59, 19.20; MS m/z: 499 (M+H)^+, 135 (100%); HRMS (FAB) calcd for C_{26}H_{38}OCl_3Si: 499.1758, found: 499.1729.

**Dichlorination of olefins 24 and 25 with a 2:1 mixture of NCS/Ph_3P (Table 2):**

**Table 2, entry 1**

(6S*,7R*)-6,7-Dichloro-2,2,3,3,17,17-hexamethyl-16,16-diphenyl-4,15-dioxa-3,16-disilaoctadecane (26)

The title compound was prepared according to the general procedure described for Table 1 using olefin 24 (52.5 mg, 0.10 mmol), Ph_3P (39.3 mg, 0.15 mmol), and NCS (40.1 mg, 0.30 mmol). (Reaction time: 5 min) Purification by flash silica gel column chromatography (n-hexane) gave chloride 26 (45.9 mg, 77%) as a colorless oil.

**Dichloride 26:** IR (neat) ν 2930, 1111, 837, 700 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.68-7.66 (m, 4H), 7.44-7.36 (m, 6H), 4.22 (m, 1H), 4.08-3.98 (m, 2H), 3.89 (dd, 1H, J = 10.8, 5.2 Hz), 3.65 (t, 2H, J = 6.4 Hz), 1.96 (m, 1H), 1.78 (m, 1H), 1.59-1.52 (m, 2H), 1.41-1.20 (m, 8H), 1.05 (s, 9H), 0.91 (s, 9H), 0.09 (s, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 135.57, 134.13, 129.47, 127.55, 65.33, 64.62, 63.90, 62.09, 33.77, 32.49, 29.17, 28.98, 26.86, 25.86, 25.78, 25.66, 19.21, 18.27, -5.46; MS m/z: 595 (M+H)^+, 73 (100%); HRMS (FAB) calcd for C_{32}H_{53}O_{2}Cl_2Si_2: 595.2961, found: 595.2958.

**Table 2, entry 2**

(2S*,3R*)-10-((tert-Butyldiphenylsilyl)oxy)-2,3-dichlorodecyl pivalate (27) and (2R*,3R*)-10-((tert-Butyldiphenylsilyl)oxy)-1,3-dichloro-2-yl pivalate (28)

The title compounds were prepared according to the general procedure described for Table 1 using olefin 25 (99.0 mg, 0.20 mmol), Ph_3P (78.7 mg, 0.30 mmol), and NCS (80.1 mg, 0.60 mmol). (Reaction time: 5 min) Purification by flash silica gel column chromatography (toluene/n-hexane 1:4) gave dichlorides 27 (76.0 mg, 66%), and 28
(14.0 mg, 12%), both as colorless oils. **Dichloride 27:** IR (neat) ν 2932, 1738, 1150, 1111, 702 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.69-7.67 (m, 4H), 7.45-7.37 (m, 6H), 4.53 (dd, 1H, J = 12.0, 3.6 Hz), 4.41 (dd, 1H, J = 12.0, 6.0 Hz), 4.18 (m, 1H), 4.08 (dt, 1H, J = 7.2, 3.2 Hz), 3.66 (t, 2H, J = 6.4 Hz), 2.04 (m, 1H), 1.83 (m, 1H), 1.58-1.53 (m, 2H), 1.44-1.20 (m, 8H), 1.24 (s, 9H), 1.06 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 177.90, 135.55, 134.10, 129.48, 127.55, 65.15, 63.88, 62.18, 1.09, 38.90, 34.64, 32.46, 29.11, 28.88, 27.10, 27.01, 26.85, 25.64, 19.20; MS m/z: 587 (M+Na)⁺, 57 (100%); HRMS (FAB) calcd for C₃₁H₄₆O₃Cl₂SiNa (M+Na)⁺: 587.2491, found: 587.2482. **Dichloride 28:** IR (neat) ν 2932, 1736, 1142, 1111, 702 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.67-7.66 (m, 4H), 7.44-7.36 (m, 6H), 5.07 (m, 1H), 4.14 (m, 1H), 3.90 (dd, 1H, J = 12.0, 5.2 Hz), 3.79 (dd, 1H, J = 12.0, 3.2 Hz), 3.65 (t, 2H, J = 6.4 Hz), 1.79 (m, 1H), 1.65 (m, 1H), 1.59-1.52 (m, 2H), 1.42-1.21 (m, 8H), 1.24 (s, 9H), 1.05 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 177.31, 135.56, 134.12, 129.49, 127.56, 74.21, 63.89, 60.25, 43.36, 39.01, 33.53, 32.48, 29.11, 28.95, 27.02, 26.86, 25.84, 25.64, 19.21; MS m/z: 587 (M+Na)⁺, 57 (100%); HRMS (FAB) calcd for C₃₁H₄₆O₃Cl₂SiNa (M+Na)⁺: 587.2491, found: 587.2467.

**References**