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Supporting Information

for

Metal-free synthesis of unsymmetric bis(thioesters)

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1. General methods and chemicals

All syntheses and catalytic tests were carried out under dry argon, using standard Schlenk-line and vacuum techniques. ¹H NMR and ¹³C NMR spectra were recorded in CD_2Cl_2 or $CDCl_3$ on a Varian 400 operating at 402.6 and 101.2 MHz, respectively. GC-MS analyses were performed on a Varian Saturn 2100T equipped with a DB-1 capillary column (30 m in length and 0.25 mm in internal diameter) and an ion trap detector. Mass spectroscopic analyses were performed using Synapt G2-S HDMS (Waters) mass spectrometer equipped with the Electrospray ion source and quadrupole-Time-of-flight mass analyzer with the resolving power FWHM 38000 using methanol as a solvent. The Capillary Voltage was set to 4.5 kV, the sampling was set 40 and the source temperature was 120°C. In the ESI-MS spectra sodiated and potassiated ions were observed as the most abundant ions. Additionally protonated ion were present in some spectra with different intensities. Thin layer chromatography (TLC) was conducted on plates coated with a 250 μ m thick silica gel layer and column chromatography was performed on silica gel 60 (70–230 mesh).

NHC carbene precursor was prepared according to literature procedures.¹ All the other reagents were commercially available and used as received. The solvents were dried over CaH₂ prior to use and stored over 4Å molecular sieves under argon. Dichloromethane was additionally passed through an alumina column and degassed by repeated freeze-pump-thaw cycles.

2. Unsymmetric thioesterification of α , β -unsaturated aldehydes with dithiols

Method 1

An oven-dried 5 mL glass reactor equipped with a magnetic stirring bar was charged with NHC carbene **NHC-1** (149.6 mg, 1.59×10^{-4} mol) in the glovebox. Then acetone (0.5 mL), dithiol **T**₁₋₄ (7.94×10⁻⁴ mol), aldehyde **A** (7.94×10⁻⁴ mol), aldehyde **B** (7.94×10⁻⁴ mol) and internal standard (decane or dodecane, 20 µL) were added. The reaction mixture was stirred at 40 °C for 24h. Reaction course was monitored by GC-MS.

Method 2

An oven-dried 5 mL glass reactor equipped with a magnetic stirring bar was charged under argon with NHC carbene precursor **NHC** (155.3 mg, 1.59×10^{-4} mol), KHMDS (38.0 mg, 1.91×10^{-4} mol) and acetone (0.5 mL). The reaction mixture was stirred at RT and after 30 minutes dithiol **T**₁₋₄ (7.94×10⁻⁴ mol), aldehyde **A** (7.94×10⁻⁴ mol), aldehyde **B** (7.94×10⁻⁴ mol) and internal standard (decane or dodecane, 20 µL) were added. The reaction mixture was stirred at 40 °C for 24h. Reaction course was monitored by GC-MS.

3. General procedure for the synthesis of bis(thioesters) (P1-P17)

A flame-dried glass reactor equipped with a magnetic stirring bar and connected to the gas and vacuum line was charged with the NHC carbene precursor **NHC** (233.5 mg, 2.39×10^{-4} mol), KHMDS (57.1 mg, 2.86×10^{-4} mol) and acetone (1 mL) under argon. After 30 minutes of vigorous stirring the solution at RT, dithiol **T**₁₋₄ (1.19 × 10⁻³ mol) and two various of aldehydes **A**, **B** (1.19 × 10⁻³ mol) were added. The reaction mixture was stirred at 40 °C until a full conversion of the substrates was detected by GC-MS. All the products were purified by column chromatography on silica gel using dichloromethane or a 1:1 v/v mixture of *n*-hexane and ethyl acetate as eluents. Evaporation of the solvents afforded analytically pure compounds.

	¹ H NMR (400 MHz, CDCl ₃ , ppm): 2.94-3.04 (m, 8H, CH_2), 3.80
	(s, 3H, OCH ₃), 6.84-6.87 (m, 2H, Ph), 7.11-7.15 (m, 2H, Ph),
	7.21-7.25 (m, 3H, Ph), 7.30-7.34 (m, 2H, Ph), 7.42 (s, 4H, -S-
	C ₆ H ₄ -S-); ¹³ C NMR (100 MHz, CDCl ₃ , ppm): 30.50 (<i>C</i> H ₂), 31.31
P1	(CH ₂), 45.22 (CH ₂), 45.54 (CH ₂), 55.22 (OCH ₃), 113.95, 126.43,
	128.33, 128.55, 129.30, 131.76, 134.70, 139.72, 158.18,
	195.69 (<i>C</i> O), 195.77 (<i>C</i> O); MS (ESI+): m/z 456 [M⁺+Na].
	¹ H NMR (400 MHz, CD ₂ Cl ₂ , ppm): 2.96-3.03 (m, 8H, CH ₂),
	6.04-6.09 (m, 1H, CH from furan), 6.27-6.33 (m, 1H, CH from
0	furan), 7.19-7.23 (m, 3H, Ph and furane), 7.28-7.34 (m, 3H,
S-C-S-C-S	Ph), 7.39-7.77 (m, 4H, Ph); ¹³ C NMR (100 MHz, CD ₂ Cl ₂ , ppm):
	24.01 (CH ₂), 31.61 (CH ₂), 42.22 (CH ₂), 45.55 (CH ₂), 106.03,
P2	110.60, 126.71, 128.71, 128.85, 135.22, 135.23, 140.32,
	141.73, 153.86, 195.73 (CO), 196.11 (CO); MS (ESI+): m/z 419
	[M ⁺ +Na].
	¹ H NMR (400 MHz, CD ₂ Cl ₂ , ppm): 2.92-2.99 (m, 8H, CH ₂), 3.76
	(s, 3H, OCH ₃), 6.81-6.85 (m, 3H, Ph), 6.97-7.02 (m, 1H, Ph),
O OMe	7.10-7.14 (m, 3H, Ph), 7.17-7.21 (m, 1H, Ph), 7.38-7.44 (m, 4H,
S-C-S-C-S	Ph); ¹³ C NMR (100 MHz, CD ₂ Cl ₂ , ppm): 30.79 (<i>C</i> H ₂), 30.81
	(CH ₂), 45.55 (CH ₂), 45.87 (CH ₂), 55.52 (OCH ₃), 114.17, 115.52
P3	(d, <i>J</i> = 21.3 Hz), 129.67, 130.28 (d, <i>J</i> = 7.9 Hz), 132.24, 135.21,
	158.61, 161.89 (d, J = 243.5 Hz), 196.03 (CO), 196.22 (CO); MS
	(ESI+): m/z 477 [M⁺+Na].
	¹ H NMR (400 MHz, CD ₂ Cl ₂ , ppm): 0.89-0.93 (m, 3H, CH ₃),
	1.31-1.38 (m, 4H, CH ₂), 1.67-1.74 (m, 2H CH ₂), 2.64-2.69 (m,
	2H, CH ₂), 2.93-3.07 (m, 4H, CH ₂), 7.18-7.25 (m, 3H, Ph),
	7.28-7.33 (m, 2H, Ph), 7.37-7.48 (m, 4H, Ph); ¹³ C NMR (100
P4	MHz, CD ₂ Cl ₂ , ppm): 14.0 (<i>C</i> H ₃), 22.66 (<i>C</i> H ₂), 25.59 (<i>C</i> H ₂), 31.40
	(CH ₂), 31.61 (CH ₂), 44.15 (CH ₂), 45.55 (CH ₂), 126.71, 127.63,
	128.71, 128.84, 135.18, 135.22, 135.41, 140.32, 196.10 (<i>C</i> O),
	196.92 (<i>C</i> O); MS (ESI+): m/z 395 [M ⁺ +Na].

4. Analytical data of isolated products

	¹ H NMR (400 MHz, CD ₂ Cl ₂ , ppm): 2.90 (s. 6H. NCH ₃).
	2.91-2.97 (m. 8H. CH2). 3.77 (s. 3H. OCH3). 6.68 (d. 2H.
	$I_{\mu\nu} = 8.7 \text{ Hz} \text{ Ph} + 6.84 \text{ (d} 2 \text{ H} I_{\mu\nu} = 8.7 \text{ Hz} \text{ Ph} + 7.07 \text{ (d} 2 \text{ Hz})$
	$J_{mn} = 8.7 H_2$, H_1 , 0.04 (d, $2H_1$, $J_{mn} = 0.7 H_2$, H_1 , 7.07 (d, $2H_1$, $J_{mn} = 8.7 H_2$, H_1 , 7.07 (d, $2H_1$, $J_{mn} = 8.7 H_2$, H_1 , 7.07 (d, $2H_1$, $J_{mn} = 8.7 H_2$, H_1 , 7.07 (d, $2H_1$, $J_{mn} = 8.7 H_2$, H_1 , 7.07 (d, $2H_1$, $J_{mn} = 8.7 H_2$, H_1 , 7.07 (d, $2H_1$, $J_{mn} = 8.7 H_2$, H_1 , 7.07 (d, $2H_1$, $J_{mn} = 8.7 H_2$, H_1 , 7.07 (d, $2H_1$, $J_{mn} = 8.7 H_2$, H_1 , 7.07 (d, $2H_1$, $J_{mn} = 8.7 H_2$, H_1 , T_2 , T_1 , T_2 , T_1 , T_2 , T_2 , T_2 , T_1 , T_2
	$J_{HH} = 0.7 \text{ Hz}, \text{ HI}, 7.13 (0, 211, 3_{HH} = 0.7 \text{ Hz}, \text{ HI}, 7.42 (0, 3, 41), 0.000 \text{ J}$
P5	PII), CINIK (100 MHz, CD_2CI_2 , ppiii). 50.81 (b) 5, CH_2), 40.87
	(NCH_3) , 45.80 (CH_2) , 40.13 (CH_2) , 55.50 (OCH_3) , 113.12,
	114.17, 129.23, 129.75, 132.24, 135.19, 135.22, 149.82,
	158.60, 196.23 (CO), 196.37 (CO); MS (ESI+): m/z 502
	[M⁺+Na].
	¹ H NMR (400 MHz, CD ₂ Cl ₂ , ppm): 2.80-2.90 (m, 6H, CH ₂), 2.93-
O S S	2.96 (m, 2H, CH ₂), 3.01 (s, 4H, SCH ₂), 3.75 (s, 3H, OCH ₃), 6.78-
	6.82 (m, 2H, Ph), 7.07-7.10 (m, 2H, Ph),
MeO	7.17-7.21 (m, 3H, Ph), 7.24-7.30 (m, 2H, Ph); ¹³ C NMR (100
P6	MHz, CD ₂ Cl ₂ , ppm): 29.05 (CH ₂), 29.09 (CH ₂), 30.76 (CH ₂),
	31.57 (CH ₂), 46.70 (CH ₂), 46.01 (CH ₂), 55.49 (OCH ₃), 114.10,
	126.60, 128.28, 128.64, 128.73, 128.78, 129.58, 132.40,
	140 49 158 51 197 99 (CO) 198 08 (CO): MS (FSI+): m/z 429
	[M ⁺ +K]
	¹ H NMR (400 MHz CD ₂ Cl ₂ nnm)· 2 82-2 86 (m 4H CH ₂) 2 91-
	$2.96 \text{ (m } AH CH_2\text{)} = 3.00 \text{ (s } AH SCH_2\text{)} = 6.92-7.01 \text{ (m } 2H \text{ Ph})$
∩ √F	$7.11_{-7.17}$ (m /H Pb) $7.22_{-7.26}$ (m 2H Pb) 1^{-3} C NMP (100
S S S	$7.11^{-7.11}$ (iii, 4ii, Fii), $7.25^{-7.20}$ (iii, 2ii, Fii), C NiNK (100
	(C_2) $(C_2$
P7	45.39 (CH ₂), 45.00 (CH ₂), 111.35, 115.50, 128.84, 130.10,
	130.23, 132.26, 136.24 (d, $J = 3.1$ Hz), $139.07, 160.62, 163.04, 139.07, 160.62, 163.04, 163.04$
	197.74 (CO), 197.83 (CO); MS (ESI+): m/z 433 [M ⁺ +Na].
	¹ H NMR (400 MHz, CD ₂ Cl ₂ , ppm): 2.80-2.85 (m, 4H, CH ₂), 2.88-
- ^	2.95 (m, 4H, CH ₂), 3.00 (s, 4H, S CH ₂), 3.75 (s, 3H, OCH ₃), 3.81
	(s, 3H, OCH ₃), 6.79-6.86 (m, 4H, Ph), 7.07-7.11 (m, 3H, Ph),
Mag O OMe	7.16-7.21 (m, 1H, Ph); ¹³ C NMR (100 MHz, CD ₂ Cl ₂ ,): 26.85
NIEO P	(CH ₂), 29.07 (CH ₂), 30.77 (CH ₂), 33.16 (CH ₂), 44.06 (CH ₂), 46.01
FO	(CH ₂), 55.49 (br s, OCH ₃), 110.56, 114.12, 120.65, 128.01,
	128.58, 129.58, 130.21, 132.40, 157.85, 158.83, 198.06 (<i>C</i> O),
	198.34 (<i>C</i> O); MS (ESI+): m/z 457 [M⁺+K].
	¹ H NMR (400 MHz, CD ₂ Cl ₂ , ppm): 2.78-2.86 (m, 8H, CH ₂), 2.89
	(s, 6H, NCH ₃), 3.00-3.02 (m, 4H, SCH ₂), 3.82 (s, 3H, OCH ₃),
	6.64-6.67 (m, 2H, Ph), 6.83-6.88 (m, 2H, Ph), 7.02-7.04 (m, 2H,
	Ph), 7.09-7.13 (m. 1H. Ph), 7.17-7.21 (m. 1H. Ph); ¹³ C NMR
Moo	(100 MHz, CD ₂ Cl ₂ , ppm): 26.86 (CH ₂), 29.09 (CH ₂), 30.75 (CH ₂),
P9	$40.86 (NCH_2) 44.07 (CH_2) 46.27 (CH_2) 55.50 (OCH_2) 110.55$
	113 08 120 64 128 01 128 17 128 59 129 15 130 21
	113.00, 120.04, 120.01, 120.17, 120.05, 125.15, 150.21,
	[M ⁺ +H]
	1 H NMR (400 MHz CD ₂ Cl ₂ nnm)· 2.82-2.98 (m. 8H CH ₂)
0	2 99-3 05 (m AH CH_{a}) 7 06-7 21 (m 5H Dh) 7 25-7 42 (m
Br	2.55 5.05 (iii, 4ii, Cii2), 7.00-7.21 (iii, 5fi, Fii), 7.25-7.42 (iii, 4H, ph), 13 C NIMP (100 MHz CD CL, ppm), 20.07 (CU), 20.00
	(C_1) 21 FE (C_1) AF 20 (C_1) AF CO (C_1) A5 20 20 (C_2)
P10	$(L\Pi_2)$, 51.55 $(L\Pi_2)$, 45.29 $(L\Pi_2)$, 45.08 $(L\Pi_2)$, 120.29, 120.60,
	128.03, 128.77, 130.54, 131.79, 139.55, 140.47, 197.69 (CO),
	197.94 (CO); MS (ESI+): m/z 477 [M⁺+K].

Br P11	¹ H NMR (400 MHz, CD ₂ Cl ₂ , ppm): 2.50-2.87, 3.03-3.12 (m, 12H, CH ₂), 2.91 (s, 6H, NCH ₃), 3.50-3.60 (m, 8H, CH ₂), 6.69-6.76 (m, 2H, Ph), 7.00-7.17 (m, 4H, Ph), 7.33-7.56 (m, 2H, Ph); ¹³ C NMR (100 MHz, CD ₂ Cl ₂ , ppm): 24.79 (CH ₂), 29.08 (CH ₂), 29.18 (CH ₂), 30.96 (CH ₂), 31.15 (CH ₂), 41.43 (NCH ₃), 45.46 (CH ₂), 46.33 (CH ₂), 70.23 (CH ₂ O),70.71 (CH ₂ O), 113.83, 120.43, 129.42, 130.73, 131.95, 139.82, 149.38, 198.21 (CO), 198.70 (CO); MS (ESI+): m/z 569 [M ⁺ +H].
MeO P12	¹ H NMR (400 MHz, CD ₂ Cl ₂ , ppm): 2.82-2.96 (m, 8H, CH ₂), 3.04- 3.10 (m, 4H, CH ₂), 3.53-3.59 (m, 8H, CH ₂), 3.76 (s, 3H, OCH ₃), 3.82 (s, 3H, OCH ₃), 6.80-6.88 (m, 4H, Ph), 7.08-7.13 (m, 3H, Ph), 7.16-7.22 (m, 1H, Ph); ¹³ C NMR (100 MHz, CD ₂ Cl ₂ , ppm): 26.84 (CH ₂), 28.81 (CH ₂), 28.88 (CH ₂), 30.77 (CH ₂), 44.04 (CH ₂), 45.96 (CH ₂), 55.42 (br s, OCH ₃), 70.00 (CH ₂ O), 70.06 (CH ₂ O), 70.50 (CH ₂ O), 110.50, 114.06, 120.60, 127.95, 128.59, 129.54, 130.15, 132.42, 157.79, 158.45, 198.31 (CO), 198.60 (CO); MS (ESI+): m/z 529 [M ⁺ +Na].
MeO P13	¹ H NMR (400 MHz, CD ₂ Cl ₂ , ppm): 2.78-2.89 (m, 6H, CH ₂), 2.90 (s, 6H, NCH ₃), 3.06-3.10 (m, 4H, CH ₂), 3.50-3.63 (m, 10H, CH ₂), 3.76 (s, 3H, OCH ₃), 6.67-6.70 (m, 1H, Ph), 6.82-6.84 (m, 2H, Ph), 7.02-7.09 (m, 2H, Ph), 7.10-7.14 (m, 3H, Ph); ¹³ C NMR (100 MHz, CD ₂ Cl ₂ , ppm): 26.86 (CH ₂), 28.89 (CH ₂), 30.75 (CH ₂), 30.78 (CH ₂), 40.93 (NCH ₃), 45.98 (CH ₂), 46.21 (CH ₂), 55.43 (OCH ₃), 70.01 (CH ₂ O), 70.04 (CH ₂ O), 70.51 (CH ₂ O), 113.18, 114.07, 129.15, 129.55, 129.58, 132.44, 158.46, 198.34 (CO), 198.51 (CO); MS (ESI+): m/z 520 [M ⁺ +H].
P14	¹ H NMR (400 MHz, CD ₂ Cl ₂ , ppm): 2.82-2.89 (m, 4H, CH ₂), 2.91- 2.98 (m, 4H, CH ₂), 3.04-3.08 (m, 4H, CH ₂), 3.52-3.57 (m, 8H, CH ₂), 3.82 (s, 3H, OCH ₃), 6.82-6.87 (m, 2H, Ph), 7.08-7.12 (m, 1H, Ph), 7.16-7.21 (m, 4H, Ph), 7.25-7.29 (m, 2H, Ph); ¹³ C NMR (100 MHz, CD ₂ Cl ₂ , ppm): 26.86 (CH ₂), 26.83 (CH ₂), 28.92 (CH ₂), 31.60 (CH ₂), 44.03 (CH ₂), 45.67 (CH ₂), 55.47 (OCH ₃), 70.01 (CH ₂ O), 70.08 (CH ₂ O), 70.53 (CH ₂ O), 110.52, 120.61, 126.56, 127.96, 128.62, 128.75, 130.17, 140.54, 157.82, 198.24 (CO), 198.62(CO); MS (ESI+): 499 [M ⁺ +Na].
MeO P15	¹ H NMR (400 MHz, CDCl ₃ , ppm): 2.91-2.94 (m, 4H, CH ₂), 2.94-3.03 (m, 4H, CH ₂), 3.76 (s, 3H, OCH ₃), 6.81-6.84 (m, 2H, Ph), 7.10-7.13 (m, 2H, Ph), 7.17-7.23 (m, 4H, Ph), 7.26-7.33 (m, 6H, Ph), 7.36-7.39 (m, 3H, Ph); ¹³ C NMR (100 MHz, CDCl ₃ , ppm): 30.80 (CH ₂), 31.60 (CH ₂), 45.43 (CH ₂), 45.75 (CH ₂), 55.51 (OCH ₃), 114.15, 126.69, 127.36, 127.42, 128.69, 128.83, 129.64, 130.54, 131.70, 132.26, 133.06, 135.45, 135.55, 137.26, 137.33, 140.35, 158.59, 196.47 (CO), 196.55 (CO); MS (ESI+): m/z 567 [M ⁺ +Na].

	¹ H NMR (400 MHz, CD ₂ Cl ₂ , ppm): 2.89-2.98 (m, 4H, CH ₂),
	2.99-3.07 (m, 4H, CH ₂), 3.77 (s, 3H, OCH ₃), 6.07 (dd, 1H,
	$J_{\rm HH}$ = 3.2, 0.7 Hz, CH from furan), 6.30 (dd, 1H,
MeO P16	J _{HH} = 3.2, 1.9 Hz, CH from furan), 6.79-6.88 (m, 2H, Ph), 7.10-
	7.16 (m, 2H, Ph and furane), 7.30-7.40 (m, 8H, Ph); ¹³ C NMR
	(100 MHz, CD ₂ Cl ₂ , ppm): 23.97 (CH ₂), 30.78 (CH ₂), 42.08 (CH ₂),
	45.73 (CH ₂), 55.50 (OCH ₃), 105.98, 110.59, 114.14, 127.17,
	127.41, 129.63, 131.68, 131.71, 132.24, 135.54, 137.27,
	141.70, 153.88, 158.58, 196.06 (CO), 196.51 (CO); MS (ESI+):
	m/z 557 [M⁺+Na].
	¹ H NMR (400 MHz, CD ₂ Cl ₂ , ppm): 2.98-3.03 (m, 8H, CH ₂), 6.07
	(dd, 1H, J _{HH} = 3.2, 0.7 Hz, CH from furan), 6.31 (dd, 1H, J _{HH} =
	3.2, 1.9 Hz, CH from furan), 7.21-7.23 (m, 2H, Ph and furane),
	7.27-7.43 (m, 12H, Ph); ¹³ C NMR (100 MHz, CD ₂ Cl ₂ , ppm):
P17	23.98 (CH ₂), 31.59 (CH ₂), 42.09 (CH ₂), 45.42 (CH ₂), 105.99,
	110.59, 126.70, 127.21 (d, J = 1.5 Hz), 127.38 (d, J = 1.5 Hz),
	128.69, 128.83, 131.71, 135.55, 137.29 (d, <i>J</i> = 3.0 Hz), 137.37
	(d, J = 3.0 Hz), 140.33, 141.71, 153.88, 196.06 (CO), 196.43
	(<i>C</i> O); MS (ESI+): m/z 527 [M⁺+Na].

5. NMR spectra of isolated products



Figure 1. ¹H NMR (400 MHz, CDCl₃) of **P1**



Figure 3. $^1\!H$ NMR (400 MHz, CD₂Cl₂) of P2



Figure 5. ^{1}H NMR (400 MHz, CD₂Cl₂) of **P3**



Figure 7. 1 H NMR (400 MHz, CD₂Cl₂) of **P4**



Figure 9. ¹H NMR (400 MHz, CD₂Cl₂) of **P5**



Figure 10. $^{\rm 13}C$ NMR (100 MHz, $CD_2Cl_2)$ of P5



Figure 11. ¹H NMR (400 MHz, CD₂Cl₂) of **P6.** Signal at 1.50 ppm derives from water from CD₂Cl₂.



Figure 12. ^{13}C NMR (100 MHz, CD_2Cl_2) of P6



Figure 13. ^{1}H NMR (400 MHz, CD₂Cl₂) of **P7**



Figure 15. ¹H NMR (400 MHz, CD₂Cl₂) of **P8.** Signal at 1.50 ppm derives from water from CD₂Cl₂.



Figure 16. ^{13}C NMR (100 MHz, CD_2Cl_2) of P8



Figure 17. 1 H NMR (400 MHz, CD₂Cl₂) of **P9**



Figure 18. ^{13}C NMR (100 MHz, CD_2Cl_2) of P9



Figure 19. 1 H NMR (400 MHz, CD₂Cl₂) of **P10**





Figure 21. ¹H NMR (400 MHz, CD_2Cl_2) of **P11**



Figure 22. $^{\rm 13}C$ NMR (100 MHz, $CD_2Cl_2)$ of P11

Product P12



Figure 23. 1 H NMR (400 MHz, CD₂Cl₂) of **P12**



Figure 24. ^{13}C NMR (100 MHz, $CD_2Cl_2)$ of P12



Figure 25. 1 H NMR (400 MHz, CD₂Cl₂) of **P13**



Figure 26. $^{\rm 13}C$ NMR (100 MHz, $CD_2Cl_2)$ of P13

Product P14



Figure 27. 1 H NMR (400 MHz, CD₂Cl₂) of **P14**



Figure 28. $^{\rm 13}C$ NMR (100 MHz, $CD_2Cl_2)$ of P14



Figure 29. 1 H NMR (400 MHz, CD₂Cl₂) of **P15**



Figure 30. ^{13}C NMR (100 MHz, CD_2Cl_2) of P15





Figure 31. 1 H NMR (400 MHz, CD₂Cl₂) of **P16**



Figure 32. $^{\rm 13}C$ NMR (100 MHz, $CD_2Cl_2)$ of P16

Product P17



Figure 35. 1 H NMR (400 MHz, CD₂Cl₂) of **P17**



Figure 36. ^{13}C NMR (100 MHz, CD₂Cl₂) of **P17**

6. References

¹ M. Hans, J. Lorkowski, A. Demonceau, L. Delaude, *Beilstein J. Org. Chem.*, 2015, **11**, 2318–2325.