

KEYWORDS: C₂₆H₅₆O₂SiSn : PGE2LA ; lower chain; chiral

ID: 28

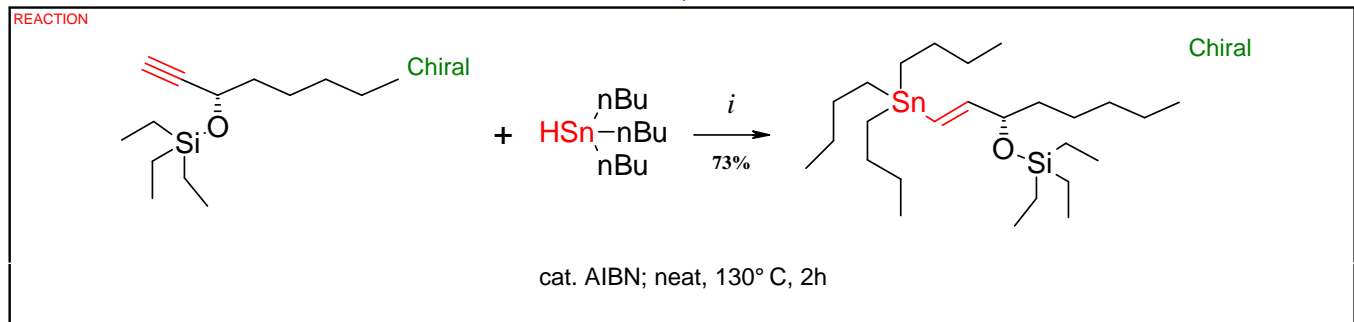
DATE: 1999.08.09

GRL-GA-28

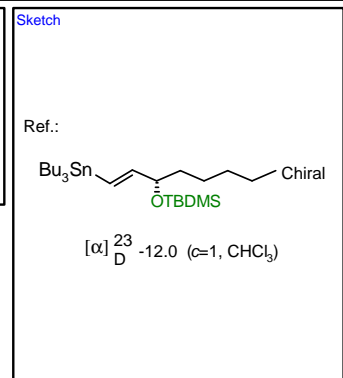
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REF: Chen S.-M. L., Schaub R. E., Grudzinskas C. V. J. Org. Chem. 1978, 43, 3450-3453
OK



RESULTS

Fr. #	Quant.	Description	Anal. Data, Identification
1	0.762	1-st drops, bp. 49-50(t/o 148)/3.7e-2mBarr	
2	0.1955	95-100(t/o 160)/3.5e-2mBarr	B: NMR
3	0.5921	rest of fraction: 100-105(t/o 160)/3.5e-2mBarr	B: NMR (traces of "1")
4	2.26	125-130(t/o 180->200)/3.3e-2mBarr	1: NMR, MS, TLC

$C_{26}H_{56}O_2SiSn$ 531.51

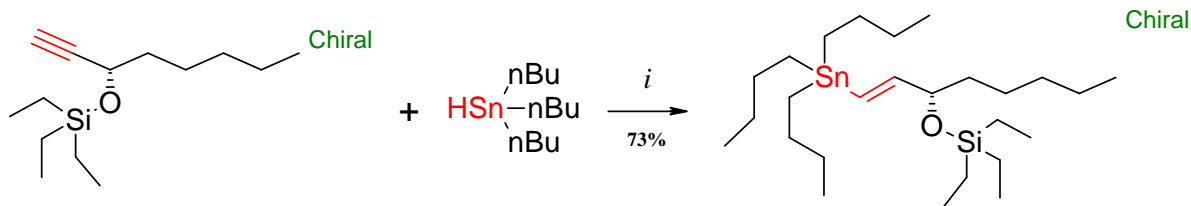
$[\alpha]_D^{20} -14.3^\circ$ (1.09, CHCl₃) (Lit. cf. Sketch: $[\alpha]_D^{20} -12.0^\circ$ (1, CHCl₃))
 colorless liquid
 bp 125-130/2.5e-2 Torr (Lit. bp. 165/5e-2 Torr)
 TLC
 MS
 NMR
 REF (racemic)
 triethyl[[(1S,2E)-1-pentyl-3-(1,1,1-tributylstannyl)-2-propenyl]oxy]silane

A neat mixture of A, B and a cat. amount of AIBN was stirred at 130° C (t/o 135°) for 2 hr under argon atmosphere. Than the resulting cloudy mixture was vacuum distilled to give fractions GRL-GA-028-01 to ..-04.

PROCEDURE

#	Formula	FW	Ratio	Scale [mol]	Quant. [mmol]	Th. Weight [mg]	Pract. Amt.: g =1 mL=0 [g] or [mL]	Excess	Volume [mL]	density [g/mL]	Molarity [mol/L]	Purity [%]	Notes
SUBSTRATES	A	C ₁₄ H ₂₈ O ₂ Si	240.46	1.000	0.005850000	5.85000	1953.74	1.95000 1	0.998	0		72.0	GRL-GA-027-01
	B	C ₁₂ H ₂₈ Sn	291.05	1.500	0.005850000	8.77500	2553.96	2.30000 0	0.975	2.360	1.082	100	Aldrich
PRODUCTS	1	C ₂₆ H ₅₆ O ₂ SiSn	531.51	1.000	0.005850000	5.85000	3109.33	2.2600	100	4.252037		73	

REACTION



cat. AIBN; neat, 130°C, 2h

73%

#	Formula	FW	Ratio	Scale [mol]	Quant. [mmol]	Th. Weight [mg]	Pract. Amt.: [g] or [mL]	g = 1 mL = 0	Excess	Volume [mL]	density [g/mL]	Molarity [mol/L]	Purity [%]	Notes
A	C ₁₄ H ₂₈ O ₂ Si	240.46	1.000	0.005850000	5.85000	1953.74	1.95000	1	0.998	0			72.0	GRL-GA-027-01
B	C ₁₂ H ₂₈ Sn	291.05	1.500	0.005850000	8.77500	2553.96	2.30000	0	0.975	2.360	1.082		100	Aldrich

#	Formula	FW	Ratio	Scale [mol]	Th. Yield [mmol]	Th. Yield [mg]	P. Yield [g]	Purity [%]	P. Yield [mmol]	P. Yield [%]	Notes
1	C ₂₆ H ₅₆ O ₂ SiSn	531.51	1.000	0.005850000	5.85000	3109.33	2.2600	100	4.252037	73	

A neat mixture of A, B and a cat. amount of AIBN was stirred at 130° C (t/o 135°) for 2 hr under argon atmosphere. Then the resulting cloudy mixture was vacuum distilled to give fractions GRL-GA-028-01 to ...04.

PROCEDURE

Fr. #	Quant.	Description	Anal. Data, Identification
1	0.762	1-st drops, bp. 49-50(t/o 148)/3.7e-2mBarr 95-100(t/o 160)/3.5e-2mBarr rest of fraction: 100-105(t/o 160)/3.5e-2mBarr 125-130(t/o 180->200)/3.3e-2mBarr	B: NMR B: NMR (traces of "1") 1: NMR, MS, TLC
2	0.1955		
3	0.5921		
4	2.26		

G
R
E
L
A
-
F
U
E

GRL-GA-
C₂₆H₅₆O₂SiSn = 531.51

G
R
E
L
A
-
F
U
E

GRL-GA-

REF: Chen S.-M. L., Schaub R. E., Grudzinskas C. V. J. Org. Chem. 1978, 43, 3450-3453

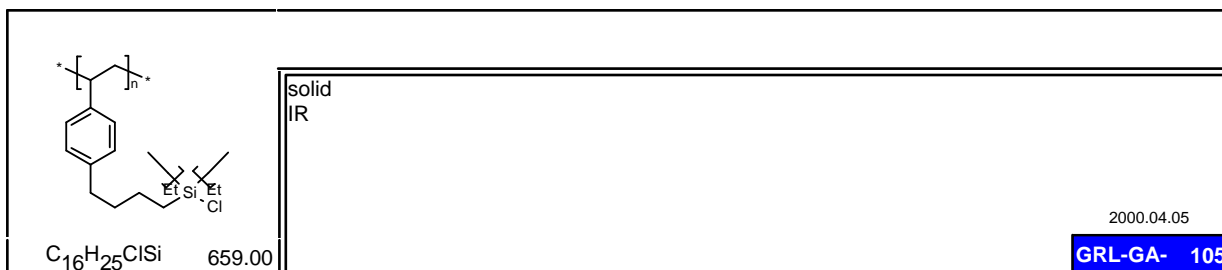
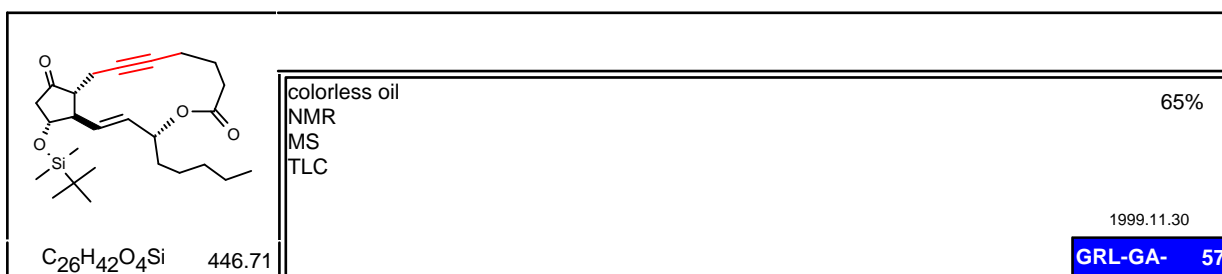
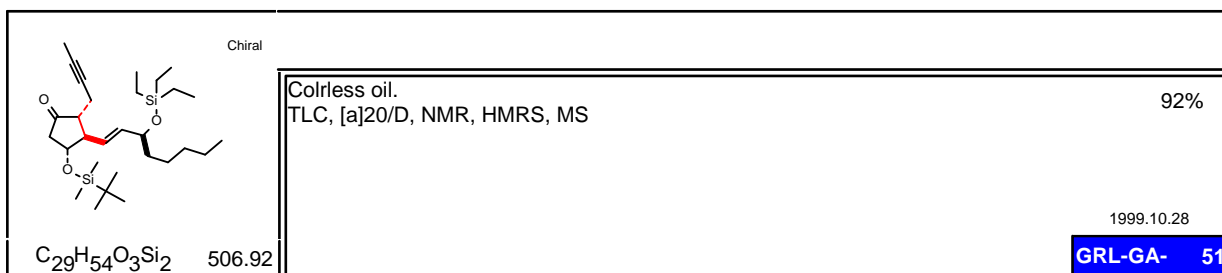
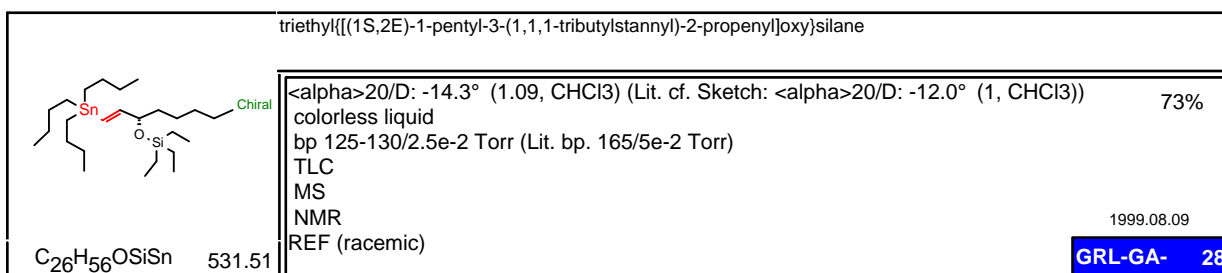
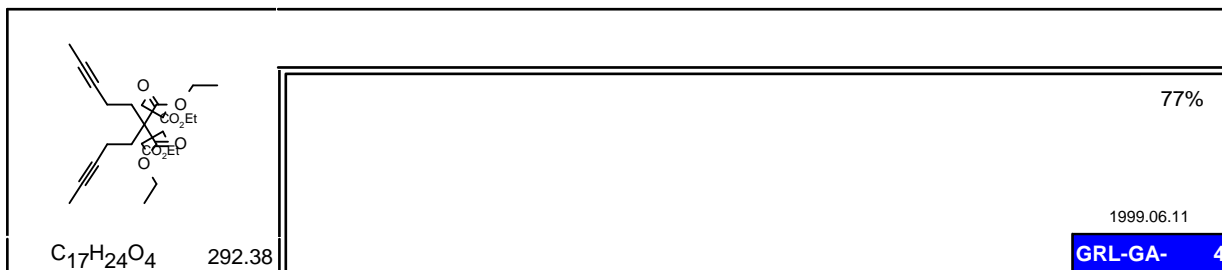
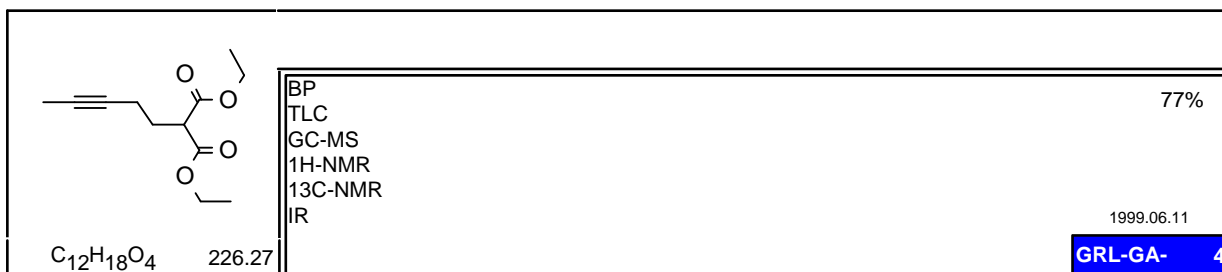
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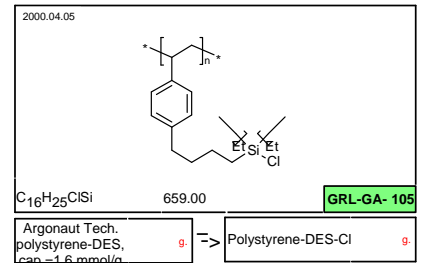
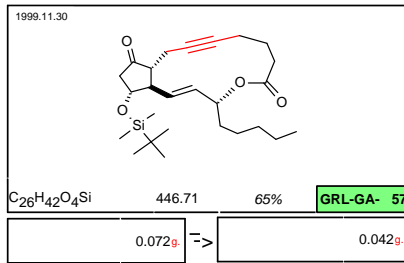
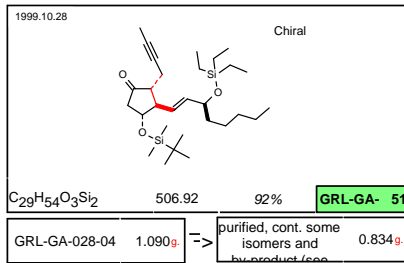
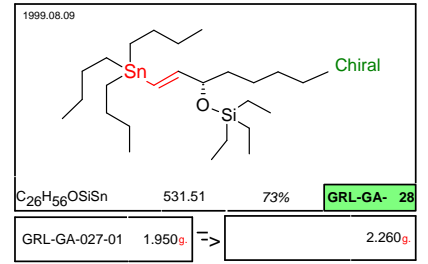
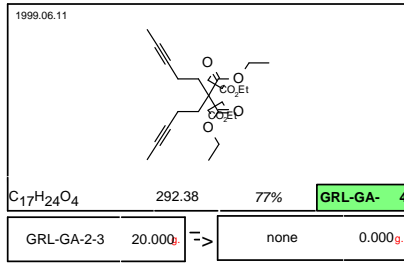
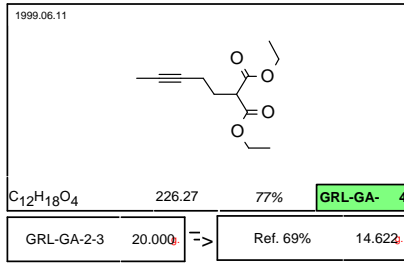
α>20/D: -14.3° (1.09, CHCl₃) (Lit. cf. Sketch: α>20/D: -12.0° (1, CHCl₃))
colorless liquid
bp 125-130/2.5e-2 Torr (Lit. bp. 165/5e-2 Torr)
TLC
MS
NMR
REF (racemic)

RESUL

Anal. Data

GDH#	Reaction	Conditions	Results
4		i) NaOEt/EtOH ; 5hr, reflux. (EtOH+Na->NaOEt)	Ref. Ansell, M.F. et al J. Chem. soc. C 1968, 217-225 OK No evidence of prod. 2 acc. to GC-MS HINT I. A lot of solid (TsONa?) is formed during the reaction. Use more ethanol (-60 mL) and better magnetic stirring element.
28		cat. AIBN; neat, 130° C, 2h	REF: Chen S.-M. L., Schaub R. E., Grudzinskas C. V. J. Org. Chem. 1978, 43, 3450-3453 OK
51		i) B+BuLi/THF; -78° , 60 min. ii) Me2Zn/PhMe; -78->0° C, 15min iii) A/THF; -78° C, 70 min. then HMPA; 5 min. iv) C/THF; -78->-40° C, 19h v) NH4Cl aq.	REF: cf. #31 NOTE 1: Acc. to HPLC (cf. #15) "A" is >85% pure (cf. Stechiometry Table of Substrates, this page) Note 2: After CC product cont. diastereoisomers: some green-spot isomer, epi-PGGr (cf. #38) and a BuLi by-product (cf. Sketch of #18). See GRL-GA-38 for more details. OK
57		PhCl (c[A] 0.01M), 80° C; 8hr	OK. Ca. 4% of substrate A was regenerated. No add. products
105		i) 0.3M 1,3-dichloro-5,5-dimethylhydantoin in CH2Cl2; r.t.; 1.5h	OK. Ref. B. M. Trost..., J. Org. Chem. 1998, 63, 4518





KEYWORDS C12H18O4 : PGE2LA , acid

ID 4

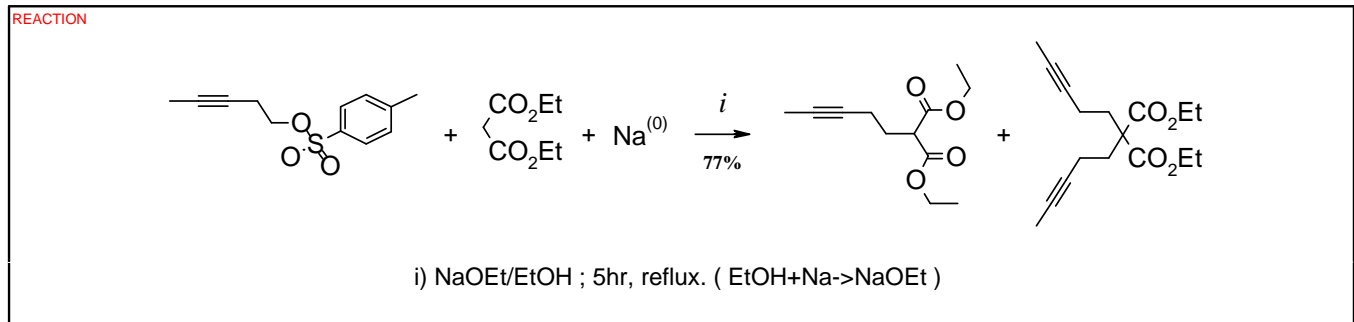
DATE 1999.06.11

GRL-GA- 4

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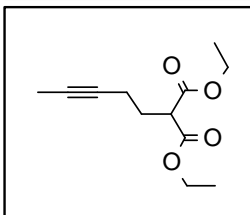
Ref. Ansell, M.F. et al J. Chem. soc. C 1968, 217-225
 OK
 No evidence of prod. 2 acc. to GC-MS
 HINT I. A lot of solid (TSONa?) is formed during the reaction. Use more ethanol (~60 mL) and better magnetic stirring element.

Sketch

RESULTS

Fr. #	Quant.	Description	Anal. Data, Identification
1	1,465g	bp. 50° /0.11 mBar (to=rt ->96°)	GC-MS : substr. B
2	9,866g	bp. 51-56° /0.11 mBar (to=96>98°)	GC-MS : substr. B
3	5,612	bp. 58-64° /0.12 mBar (to= 98->110°)	GC-MS : substr. B
4	14,6217	bp. 101-104° /0.13 mBar (to= 110->125°), prod. 1, contain a few drop of fr. 3!	GC-MS, C- H-NMR, IR, TLC : prod. 1

C₁₂H₁₈O₄ 226.27

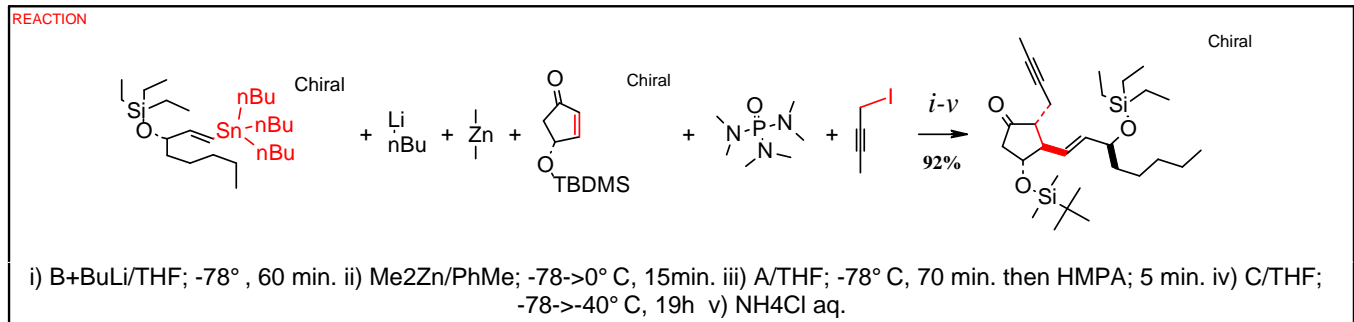


BP
 TLC
 GC-MS
 1H-NMR
 13C-NMR
 IR

Argon. A sol. of A in EtOH (anh., 30 mL) and THF (anh. 10 mL) was added during 1h to a stirred solution of B and EtONa (prepared in situ by dissolution of C in EtOH) in EtOH (30 mL). After 15 min. an additional amount of EtOH was added (30 mL, HINT I) and the mixt. was refluxed for 5 h., cooled and poured into the water (100 mL). After extraction (EtOAc, 3x50mL) the extract was washed with water (50 mL), dried (MgSO₄) and distilled to yield 2 fraction: substrate A (GRL-GA-004-01 to ...03) and the product 1 (GRL-GA-004-04)

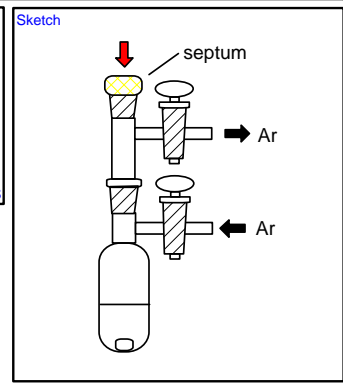
PROCEDURE

#	Formula	FW	Ratio	Scale [mol]	Quant. [mmol]	Th. Weight [mg]	Pract. Amt.: [g] or [mL]	g = 1 mL=0	Excess	Volume [mL]	density [g/mL]	Molarity [mol/L]	Purity [%]	Notes
A	C ₁₂ H ₁₄ O ₃ S	238.31	1.000	0.100000000	100.00000	23831.00	20.00000	1	0.839	0			100	GRL-GA-2-3
B	C ₇ H ₁₂ O ₄	160.17	2.000	0.100000000	200.00000	32034.00	32.48000	1	1.014	0			100	B-1568
C	Na	22.99	1.000	0.100000000	100.00000	2299.00	2.31000	1	1.005	0			100	B-2160
#	Formula	FW	Ratio	Scale [mol]	Th. Yield [mmol]	Th. Yield [mg]	P. Yield [g]	Purity [%]	P. Yield [mmol]	P. Yield [%]	PRODUCTS			
1	C ₁₂ H ₁₈ O ₄	226.27	0.839	0.100000000	83.90000	18984.05	14.6217	100	64.620586	77	Ref. 69%			
2	C ₁₇ H ₂₄ O ₄	292.38	0.839	0.100000000	83.90000	24530.68	0.0000	100	0	0	none			



REF: cf. #31
 NOTE 1: Acc. to HPLC (cf. #15) "A" is >85% pure (cf. Stechiometry Table of Substrates, this page)
 Note 2: After CC product cont. diastereoisomers: some green-spot isomer, epi-PGGr (cf. #38) and a BuLi by-product (cf. Sketch of #18). See GRL-GA-38 for more details.

OK



RESULTS

Fr. #	Quant.	Description	Anal. Data, Identification
1	0.8340	fr. 56-80, crude "1", cont. traces of 2 isomers, for synth.	TLC, [α] ₂₀ /D, NMR, HMRS, MS

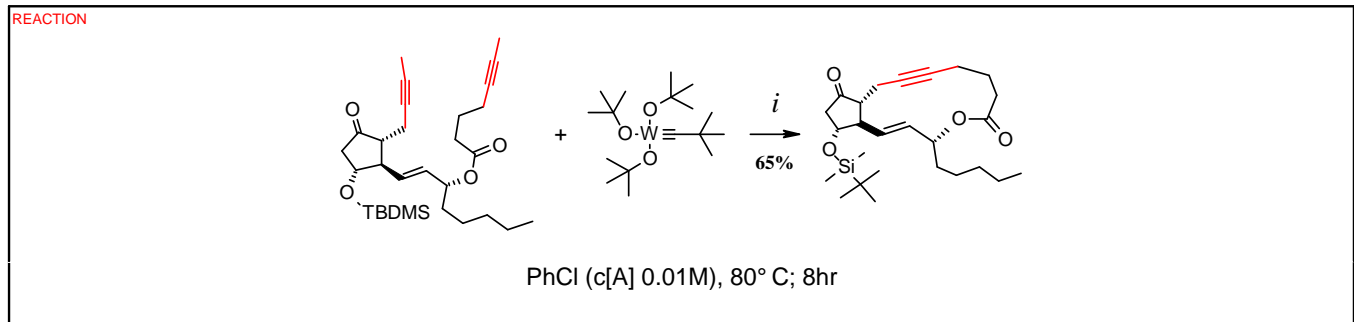
Chiral	Colorless oil. TLC, [α] ₂₀ /D, NMR, HMRS, MS	C ₂₉ H ₅₄ O ₃ Si ₂ 506.92
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Cf. #31 for details. Reaction of vinylstannane A (Note 1) with BuLi (1h @ -78° C) and then Me2Zn (15 min @ 0° C). Addition of D in THF (6mL) over a period of 70 min. (syringe pump, 5 mL-plastic syringe, 0.6-0.7cm/min). After addition of E and F the mixture was stirred for 19 hr at -40(+/-5)° C. After quench with sat. NH4Cl (3 mL) and Extrelut-workup the resulting red oil was subjected to column chromatography (PhMe:HexH:EtOAc 130:100:5 v/v) to give purified "1" (Note 2)

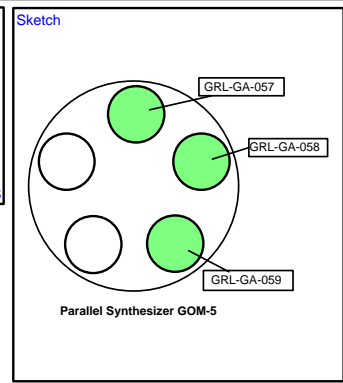
PROCEDURE

#	Formula	FW	Ratio	Scale [mol]	Quant. [mmol]	Th. Weight [mg]	Pract. Amt.: [g] or [mL]	g = 1 mL = 0	Excess	Volume [mL]	density [g/mL]	Molarity [mol/L]	Purity [%]	Notes
A	C ₂₆ H ₅₆ O ₃ SiSn	531.51	1.000	0.001850000	0.00180	1092.55	1.09020	1	0.998	0			90.0	GRL-GA-028-04
B	C ₄ H ₉ Li	64.06	1.000	0.001850000	1.85000	118.51	1.15000	0	0.995	1.156		1.60	100	1.6M in Hexanes
C	C ₂ H ₆ Zn	95.44	1.000	0.001850000	1.85000	176.56	0.95000	0	1.027	0.925		2.00	100	2M sol. in PhMe
D	C ₁₁ H ₂₀ O ₂ Si	212.37	0.970	0.001850000	1.79450	381.10	0.38400	1	1.008	0			100	GRL-GA-050-22 cryst., 94% ee
E	C ₆ H ₁₈ N ₃ OP	179.20	10.000	0.001850000	18.50000	3315.20	3.50000	0	1.087	3.219	1.030		100	
F	C ₄ H ₅ I	179.99	5.000	0.001850000	9.25000	1664.91	1.70700	1	1.025	0			100	GRL-GA-030-01

#	Formula	FW	Ratio	Scale [mol]	Th. Yield [mmol]	Th. Yield [mg]	P. Yield [g]	Purity [%]	P. Yield [mmol]	P. Yield [%]	Notes
1	C ₂₉ H ₅₄ O ₃ Si ₂	506.92	0.970	0.001850000	1.79450	909.67	0.8340	100	1.645230	92	purified, cont. some isomers and by-product (see TLC)



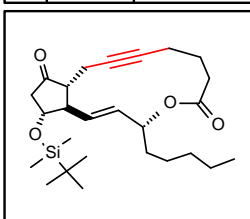
OK.
Ca. 4% of substrate A was regenerated. No add. products



RESULTS

Fr. #	Quant.	Description	Anal. Data, Identification
1	41.7mg	fr. 21-33 in CC: pure product 1	full
2	3mg	fr. 35-36 in CC: substrate A	TLC

C₂₆H₄₂O₄Si 446.71



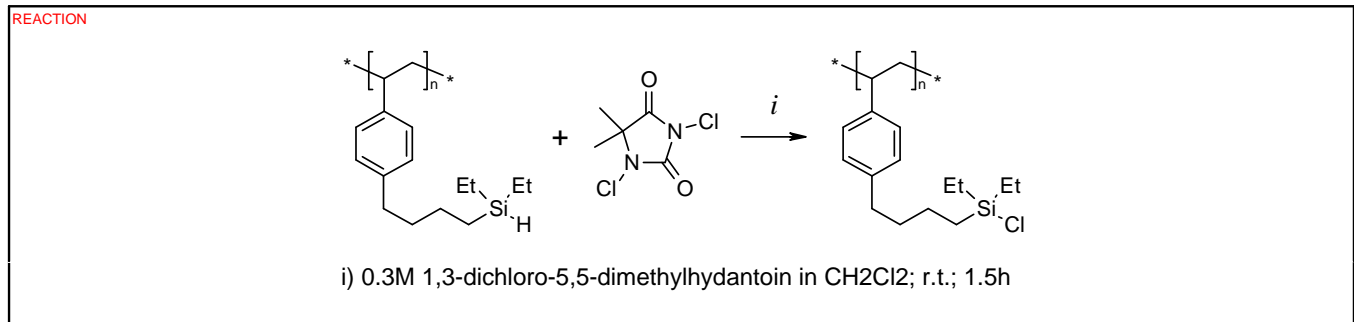
colorless oil
NMR
MS
TLC

Reaction in GOM-5. The orange-brown solution of B in dry toluene (10mL, Note 1) and dry CH₂Cl₂ (2mL) was stirred at rt for 5 min. Then a solution of A in PhMe (2x2mL) was added and the resulting solution was quickly warmed to 80 degC (t/o 85 degC, Note 2) and stirred for 22hr. The resultin mixture was cooled to rt, evaporated (on SiO₂) and chromatographed (EH 10%)

PROCEDURE

#	Formula	FW	Ratio	Scale [mol]	Quant. [mmol]	Th. Weight [mg]	Pract. Amt.: [g] or [mL]	g = 1 mL = 0	Excess	Volume [mL]	density [g/mL]	Molarity [mol/L]	Purity [%]	Notes
A	C ₃₀ H ₄₈ O ₄ Si	500.80	1.000	0.000140000	0.14000	70.11	0.07184	1	1.025	0			100	
B	C ₁₇ H ₃₆ O ₃ W	472.32	0.100	0.000140000	0.01400	6.61	0.00800	1	1.210	0			100	
#	Formula	FW	Ratio	Scale [mol]	Th. Yield [mmol]	Th. Yield [mg]	P. Yield [g]	Purity [%]	P. Yield [mmol]	P. Yield [%]	Notes			
q	C ₂₆ H ₄₂ O ₄ Si	446.71	1.025	0.000140000	0.14350	64.10	0.0417	100	0.093349	65				

PRODUCTS

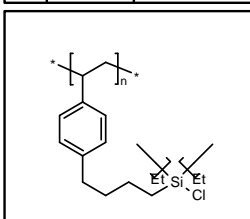


OK.
Ref. B. M. Trost..., J. Org. Chem. 1998, 63, 4518

Sketch

Fr. #	Quant.	Description	Anal. Data, Identification
1	--	solid polymer after washing	IR

RESULTS



solid
IR

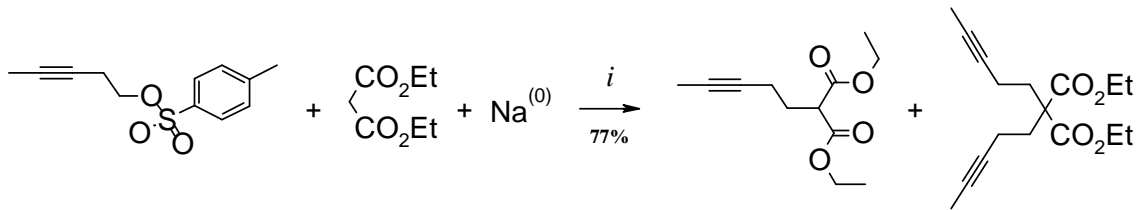
C₁₆H₂₅ClSi 659.00

To a 5 mL round-bottom flask under argon were added a silane resin A, and a 0.3M sol. of B in CH₂Cl₂. After 1.5 hr. the mixture was filtered and washed with CH₂Cl₂ (3x3mL) and a dry THF (2x3mL). The chlorinated resin (1) was used for f the next reaction (see GRL-GA-110) immediately after washing.

PROCEDURE

#	Formula	FW	Ratio	Scale [mol]	Quant. [mmol]	Th. Weight [mg]	Pract. Amt.: g = 1 mL = 0	Excess	Volume [mL]	density [g/mL]	Molarity [mol/L]	Purity [%]	Notes
A	C ₁₆ H ₂₆ Si	625.00	1.000	0.000320000	0.32000	200.00	1	0	0			100	Argonaut Tech. polystyrene-DES, cap.=1.6 mmol/g (cFW=625g/mol) Aldrich
B	C ₅ H ₆ Cl ₂ N ₂ O ₂	197.02	3.000	0.000320000	0.96000	189.14	1	0	0			100	
PRODUCTS													
#	Formula	FW	Ratio	Scale [mol]	Th. Yield [mmol]	Th. Yield [mg]	P. Yield [g]	Purity [%]	P. Yield [mmol]	P. Yield [%]	Notes		
A	C ₁₆ H ₂₅ ClSi	659.00	1.000	0.000320000	0.32000	210.88		100	0	0	Polystyrene-DES-Cl		

REACTION



i) NaOEt/EtOH ; 5hr, reflux. (EtOH+Na->NaOEt)

77%

#	Formula	FW	Ratio	Scale [mol]	Quant. [mmol]	Th. Weight [mg]	Pract. Amt.: [g] or [mL]	g = 1 mL = 0	Excess	Volume [mL]	density [g/mL]	Molarity [mol/L]	Purity [%]	Notes
A	C ₁₂ H ₁₄ O ₃ S	238.31	1.000	0.100000000	100.00000	23831.00	20.00000	1	0.839	0			100	GRL-GA-2-3
B	C ₇ H ₁₂ O ₄	160.17	2.000	0.100000000	200.00000	32034.00	32.48000	1	1.014	0			100	B-1568
C	Na	22.99	1.000	0.100000000	100.00000	2299.00	2.31000	1	1.005	0			100	B-2160

#	Formula	FW	Ratio	Scale [mol]	Th. Yield [mmol]	Th. Yield [mg]	P. Yield [g]	Purity [%]	P. Yield [mmol]	P. Yield [%]	Notes
1	C ₁₂ H ₁₈ O ₄	226.27	0.839	0.100000000	83.90000	18984.05	14.6217	100	64.620586	77	Ref. 69%
2	C ₁₇ H ₂₄ O ₄	292.38	0.839	0.100000000	83.90000	24530.68	0.0000	100	0	0	none

Argon. A sol. of A in EtOH (anh., 30 mL) and THF (anh. 10 mL) was added during 1h to a stirred solution of B and EtONa (prepared in situ by dissolution of C in EtOH) in EtOH (30 mL). After 15 min. an additional amount of EtOH was added (30 mL, HINT I) and the mixt. was refluxed for 5 h., cooled and poured into the water (100 mL). After extraction (EtOAc, 3x50mL) the extract was washed with water (50 mL), dried (MgSO₄) and distilled to yield 2 fraction: substrate A (GRL-GA-004-01 to ...03) and the product 1 (GRL-GA-004-04)

PROCEDURE

Fr. #	Quant.	Description	Anal. Data, Identification
1	1,465g	bp. 50°/0.11 mBar (to=rt ->96°)	GC-MS : substr. B GC-MS : substr. B GC-MS : substr. B GC-MS, C- H-NMR, IR, TLC : prod. 1
2	9,866g	bp. 51-56°/0.11 mBar (to=96>98°)	
3	5,612	bp. 58-64°/0.12 mBar (to= 98->110°)	
4	14.6217	bp. 101-104°/0.13 mBar (to= 110->125°), prod. 1, contain a few drops of fr. 3!	

G
R
E
L
A
F
U
E

GRL-GA-

C₁₂H₁₈O₄ = 226.27

GRL-GA-

G
R
E
L
A
F
U
E

GRL-GA-

GRL-GA-

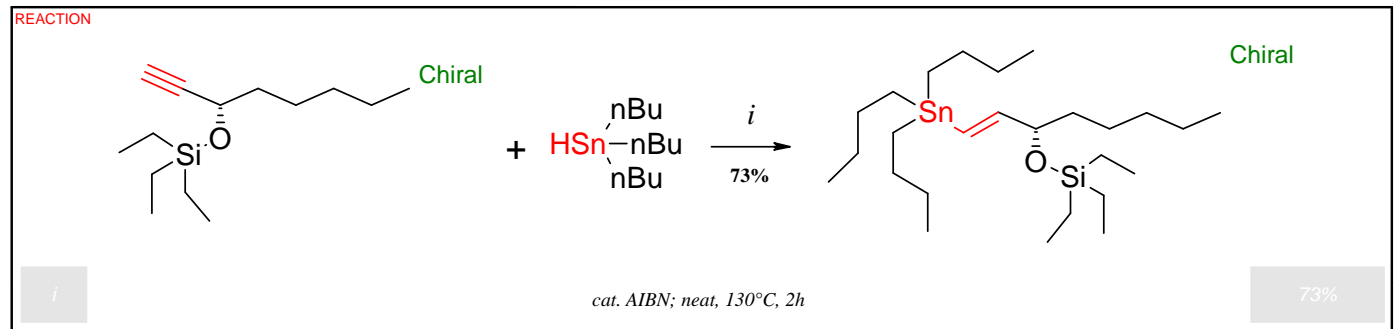
Ref. Ansell, M.F. et al J. Chem. soc. C 1968, 217-225
OK

No evidence of prod. 2 seen to GC-MS

BP
TLC
GC-MS
1H-NMR
13C-NMR
IR

RESULTS

Anal. Data



#	Formula	FW	Ratio	Scale [mol]	Quant. [mmol]	Th. Weight [mg]	Pract. Amt.: [g] or [mL]	g = 1 mL = 0	Excess	Volume [mL]	density [g/mL]	Molarity [mol/L]	Purity [%]	Notes
A	C ₁₄ H ₂₈ O ₂ Si	240.46	1.000	0.005850000	5.85000	1953.74	1.95000	1	0.998	0			72.0	GRL-GA-027-01
B	C ₁₂ H ₂₈ Sn	291.05	1.500	0.005850000	8.77500	2553.96	2.30000	0	0.975	2.360	1.082		100	Aldrich

#	Formula	FW	Ratio	Scale [mol]	Th. Yield [mmol]	Th. Yield [mg]	P. Yield [g]	Purity [%]	P. Yield [mmol]	P. Yield [%]	Notes
1	C ₂₆ H ₅₆ O ₂ SiSn	531.51	1.000	0.005850000	5.85000	3109.33	2.2600	100	4.252037	73	

A neat mixture of A, B and a cat. amount of AIBN was stirred at 130° C (t/o 135°) for 2 hr under argon atmosphere. Than the resulting cloudy mixture was vacuum distilled to give fractions GRL-GA-028-01 to ..-04.

PROCEDURE

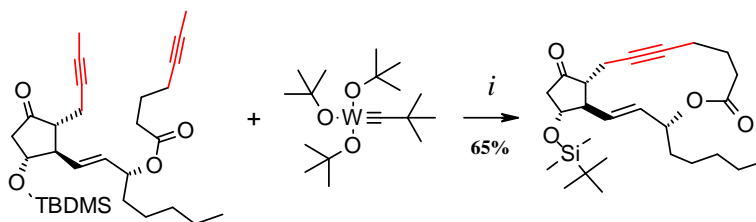
Fr. #	Quant.	Description	Anal. Data, Identification
1	0.762	1-st drops, bp. 49-50(t/o 148)/3.7e-2mBarr 95-100(t/o 160)/3.5e-2mBarr rest of fraction: 100-105(t/o 160)/3.5e-2mBarr 125-130(t/o 180->200)/3.3e-2mBarr	B: NMR B: NMR (traces of "1") 1: NMR, MS, TLC
2	0.1955		
3	0.5921		
4	2.26		

G R E L A F U E	
GRL-GA-	
C ₂₆ H ₅₆ O ₂ SiSn = 531.51	
GRL-GA-	
GRL-GA-	

G R E L A F U E	
GRL-GA-	
GRL-GA-	
GRL-GA-	

REF: Chen S.-M. L., Schaub R. E., Grudzinskas C. V. J. Org. Chem. 1978, 43, 3450-3453	RESULTS
OK	
αD: -14.3° (1.09, CHCl ₃) (Lit. cf. Sketch: αD: -12.0° (1, CHCl ₃))	Anal. Data
colorless liquid	
bp 125-130/2.5e-2 Torr (Lit. bp. 165/5e-2 Torr)	
TLC	
MS	
NMR	
REF (racemic)	

REACTION



PhCl (c[A] 0.01M), 80°C; 8hr

65%

#	Formula	FW	Ratio	Scale [mol]	Quant. [mmol]	Th. Weight [mg]	Pract. Amt.: [g] or [mL]	g = 1 mL = 0	Excess	Volume [mL]	density [g/mL]	Molarity [mol/L]	Purity [%]	Notes
A	C ₃₀ H ₄₈ O ₄ Si	500.80	1.000	0.000140000	0.14000	70.11	0.07184	1	1.025	0			100	
B	C ₁₇ H ₃₆ O ₃ W	472.32	0.100	0.000140000	0.01400	6.61	0.00800	1	1.210	0			100	

#	Formula	FW	Ratio	Scale [mol]	Th. Yield [mmol]	Th. Yield [mg]	P. Yield [g]	Purity [%]	P. Yield [mmol]	P. Yield [%]	Notes
q	C ₂₆ H ₄₂ O ₄ Si	446.71	1.025	0.000140000	0.14350	64.10	0.0417	100	0.093349	65	

Reaction in GOM-5. The orange-brown solution of B in dry toluene (10mL, Note 1) and dry CH₂Cl₂ (2mL) was stirred at rt for 5 min. Then a solution of A in PhMe (2x2mL) was added and the resulting solution was quickly warmed to 80 degC (t/o 85 degC, Note 2) and stirred for 22hr. The resultin mixture was cooled to rt, evaporated (on SiO₂) and chromatographed (EH 10%)

PROCEDURE

Fr. #	Quant.	Description	Anal. Data, Identification
1	41.7mg	fr. 21-33 in CC: pure product 1	full
2	3mg	fr. 35-36 in CC: substrate A	TLC

G
R
E
L
A
-
F
U
E

GRL-GA-
C₂₆H₄₂O₄Si = 446.71

G
R
E
L
A
-
F
U
E

GRL-GA-

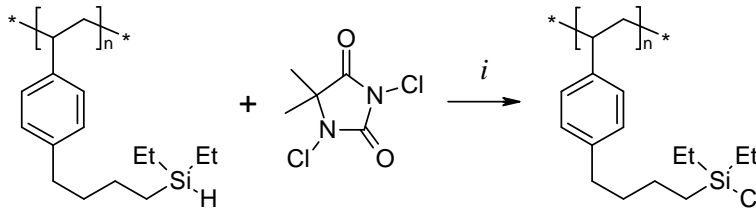
OK.
Ca. 4% of substrate A was regenerated. No add. products

colorless oil
NMR
MS
TLC

RESULTS

Anal. Data

REACTION



i) 0.3M 1,3-dichloro-5,5-dimethylhydantoin in CH₂Cl₂; r.t.; 1.5h

#	Formula	FW	Ratio	Scale [mol]	Quant. [mmol]	Th. Weight [mg]	Pract. Amt.: [g] or [mL]	g = 1 mL = 0	Excess	Volume [mL]	density [g/mL]	Molarity [mol/L]	Purity [%]	Notes
A	C ₁₆ H ₂₆ Si	625.00	1.000	0.000320000	0.32000	200.00		1	0	0			100	Argonaut Tech. polystyrene-DES, cap.=1.6 mmol/g (cFW=625g/mol)
B	C ₅ H ₆ Cl ₂ N ₂ O ₂	197.02	3.000	0.000320000	0.96000	189.14		1	0	0			100	Aldrich

#	Formula	FW	Ratio	Scale [mol]	Th. Yield [mmol]	Th. Yield [mg]	P. Yield [g]	Purity [%]	P. Yield [mmol]	P. Yield [%]	Notes
A	C ₁₆ H ₂₅ ClSi	659.00	1.000	0.000320000	0.32000	210.88		100	0	0	Polystyrene-DES-Cl

To a 5 mL round-bottom flask under argon were added a silane resin A, and a 0.3M sol. of B in CH₂Cl₂. After 1.5 hr. the mixture was filtered and washed with CH₂Cl₂ (3x3mL) and a dry THF (2x3mL). The chlorinated resin (1) was used for f the next reaction (see GRL-GA-110) immediately after washing.

PROCEDURE

Fr. #	Quant.	Description	Anal. Data, Identification
1	--	solid polymer after washing	IR

G
R
E
L
A
-
F
U
E

GRL-GA-
C₁₆H₂₅ClSi = 659.00

GRL-GA-

G
R
E
L
A
-
F
U
E

GRL-GA-

GRL-GA-

GRL-GA-

OK.
Ref. B. M. Trost..., J. Org. Chem. 1998, 63, 4518

RESULTS

solid
IR

Anal. Data

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