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## A stereoselective synthesis of 1,2-disubstituted alkenyl selenides via hydroboration-iodination of internal alkylselenoacetylenes with dicyclohexylborane

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#### Abstract

Selenoalkenyldicyclohexylboranes, prepared conveniently via hydroboration of internal alkylselenoacetylenes with dicyclohexylborane followed by iodination under basic conditions, produce cis/trans 1,2-disubstituted alkenyl selenides (1 and 2), which provide a general method for synthesis of cis/trans disubstituted alkenyl selenides containing a cyclohexyl group.

Keywords: Dicyclohexylborane; Alkylselenoacetylenes; Hydroboration; Iodination; Alkenyl selenides

#### **1. Introduction**

Many biologically active compounds occurring in nature possess the structural skeleton of trisubstituted alkenes [1-3]. Because disubstituted alkenyl selenides are synthetically equivalent to carbonyls and can be stereospecifically converted to trisubstituted alkenes by nickel-catalyzed coupling reactions with Grignard reagents [4], the high-yield, stereoselective synthesis of disubstituted alkenyl selenides is a highly desirable goal. Although the hydroboration of acetylene has been studied intensively [5], reports on this subject are still appearing [6]. However, to date there are no reported studies of hydroboration of alkylselenoacetylenes with dialkylboranes. Therefore, we now wish to report that cis/trans 1,2-disubstituted alkenyl selenides could be synthesized by hydroboration of internal alkylselenoacetylenes, followed by iodination in the presence of a base, which results in a transfer of one cyclohexyl group from a boron atom to an adjacent carbon.

### 2. Results and discussion

Zweifel et al. [7] previously reported the hydroboration-iodination of acetylene, which results in a transfer of one cyclohexyl group from boron to an adjacent carbon to give a high yield of the (Z)-alkene with high isomeric purity. However, addition of a solution of internal alkylselenoacetylene [8] to dicyclohexylborane gave the selenoalkenyldicyclohexylboranes, and successive treatment of these intermediate selenoalkenylboranes with sodium hydroxide and a solution of iodine in THF yielded a mixture of *cis/trans* disubstituted alkenyl selenides (1 and 2) (Eq. (1)). The ratio of *cis/trans* isomers, established by <sup>1</sup>H NMR measurements without separation, depends on the internal alkylselenoacetylene employed in the reaction. The results of the reaction are summarized in Table 1.

$$(c-C_{6}H_{11})_{2}BH + R^{1}Se \xrightarrow{R^{2}} R^{2}$$

$$\xrightarrow{\text{THF}} R^{1}Se \xrightarrow{H} R^{2}$$

$$\xrightarrow{\text{NaOH}/1_{2}} \xrightarrow{c-C_{6}H_{11}} \xrightarrow{H} R^{2} \xrightarrow{H} R^{2} \xrightarrow{R^{2}} R^{2}$$

$$1 \qquad 2 \qquad (1)$$

The stereochemistry of 1 and 2 was established by <sup>1</sup>H NMR measurements. One olefinic proton signal of 1 or 2 was characteristically split into one triplet with coupling contant J = 7.2 Hz, which indicates that the

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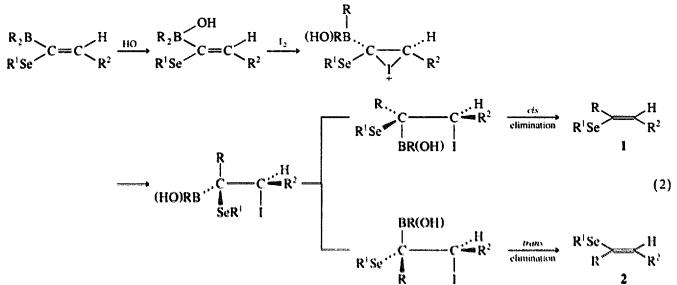
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hydroboration of internal alkylselenoacetylenes with dicyclohexylborane proceeds with strong preference for the addition of the boron atom at the carbon adjacent to the alkylseleno group. The *cis/trans* isomers could also be transformed into *trans* **3** and *cis* **4** isomers of 1-cyclohexyl-1-hexene, that are known to have unambiguous stereochemistry [9,10] and could be obtained by treatment of **1a** and **2a**, separated respectively with LiAlH<sub>4</sub> in THF with retention of the configuration [11].

$$\begin{array}{c} c - C_6 H_{11} \\ H_b \\ H_b \\ 3 \end{array} \xrightarrow{H_a} \begin{array}{c} c - C_6 H_{11} \\ H_b \\ H_b \\ H_a \end{array} \xrightarrow{H_a} \begin{array}{c} n - C_4 H_9 \\ H_a \\ H_a \end{array}$$

The ratio of isomers 1/2 depends on the size of the R<sup>1</sup> and R<sup>2</sup> of R<sup>1</sup>Se==-R<sup>2</sup>. The proportion of 1 would

be reduced as  $\mathbb{R}^2$  is altered to a bulkier group (Table 1), whereas the result is contrary with  $R^{1}$  trans formed from the methyl group to ethyl. Apparently, the bulkiness of  $R^1$  and  $R^2$  seems to promote the alteration of the ratio of 1/2. It is not clear why the hydroborationiodination of internal alkylselenoacetylenes with dicyclohexborane produces cis isomers 1. On the basis of the normally accepted mechanisms for the formation of trisubstituted alkenes, the reaction proceeds via trans addition, followed by trans elimination, and that only gives *cis* alkenes [12]. Thus, in the present case an additional consideration is that the reaction proceeds via trans addition, followed by cis and trans deboronoiodination with different steric requirements, resulting in the cis and trans stereochemistry of the two substituents from internal alkylselenoacetylenes (Eq. (2)).



In summary, our results show that the hydroboration=iodination sequence of internal alkylselenoacetylenes with dicyclohexylborane represents a very convenient stereoselective synthesis of *cis/trans* 1,2-disubstituted alkenyl selenides under mild conditions. We are presently exploring the possibilities of extending this procedure for the synthesis of disubstituted alkenyl selenides containing more complex cyclic systems.

#### 3. Experimental section

<sup>1</sup>H NMR spectra were recorded on an AZ-300 MHz with TMS as internal standard. Mass spectra were determined by a Finigan 8230 mass spectrometer. IR spectra were obtained as neat capillary cells (liquid products) on a Shimadzu IR-408 instrument. All syntheses of complexes 1 and 2 were carried out under nitrogen using standard techniques [13]. Solvents were dried, deoxygenated and distilled before use. Dicyclohexylbo-

rane and internal alkylselenoacetylene were prepared according to the literature methods, Refs. [13] and [8] respectively. The borane was made in the laboratory and standardized before use. Commercial sodium borohydride was used without purification.

## 3.1. General synthesis of cis / trans disubstituted vinylic selenides (1 and 2)

To a solution of cyclohexene (10 mmol) in THF (5 ml) was added a solution of borane (5 mmol) in THF (1.25 ml) at 0°C with stirring. The precipitate formed ( $R_2BH$ ) was stirred at 0-5°C for 1 h, and then the reaction mixture was diluted with a solution of internal alkylselenoacetylene (5 mmol) in THF (5 ml) added at 10°C. After the precipitate had dissolved, the resulting solution was stirred for an additional 30 min at room temperature. To this mixture was then added at  $-10^{\circ}C$  6 N sodium hydroxide, followed by the dropwise addition of a solution of iodine (5 mmol) in THF (3 ml) over a period of 15 min. After the reaction mixture had

warmed to room temperature, any excess iodine was decomposed by adding a small amount of aqueous sodium thiosulfate. Solvent was removed in vacuo and the residue was separated by flash chromatography on a 3 ft  $\times$  1 in column (100–150 mcsh) by elution with pentane to give 1 and 2 respectively.

#### 3.1.1. cis-1-Methylseleno-1-cyclohexyl-1-hexene (1a)

Oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>,  $\delta$  ppm): 6.27 (t, 1H, J = 7Hz); 2.47 (s, 3H); 1.9–2.33 (m, 3H); 0.95–1.58 (m, 17H). IR  $\nu$ (cm<sup>-1</sup>): 1630, 807. MS m/z: 258 (M - 1). Anal. Found: C, 60.41; H, 9.31. C<sub>13</sub>H<sub>24</sub>Se Calc.: C, 60.22; H, 9.33%.

3.1.2. trans-1-Methylseleno-1-cyclohexyl-1-hexene (2a) Oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>,  $\delta$  ppm): 6.81 (t, 1H, J = 7 Hz); 2.43 (s, 3H); 1.90–2.31 (m, 3H); 0.95–1.57 (m, 17H). IR  $\nu$ (cm<sup>-1</sup>): 1628, 796. MS m/z: 258 (M – 1). Anal. Found: C, 60.31; H, 9.30. C<sub>13</sub>H<sub>24</sub>Se Calc.: C, 60.22; H, 9.33%.

#### 3.1.3. cis-1-Ethylseleno-1-cyclohexyl-1-hexene (1b)

Oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>,  $\delta$  ppm): 6.25 (t, 1H, J = 7Hz); 2.81 (q, 2H, J = 7.2 Hz); 1.93–2.33 (m, 3H); 0.94–1.84 (m, 20H). IR  $\nu$ (cm<sup>-1</sup>): 1631, 805. MS m/z: 272 (M - 1). Anal. Found: C, 61.54; H, 9.71. C<sub>14</sub>H<sub>26</sub>Se Calc.: C, 61.52; H, 9.59%.

#### 3.1.4. trans-1-Ethylseleno-1-cyclohexyl-1-hexene (2b)

Oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>,  $\delta$  ppm): 6.89 (t, 1H, J = 7Hz); 2.80 (q, 2H, J = 7.2 Hz); 1.93–2.34 (m, 3H); 0.95–1.85 (m, 20H). IR  $\nu$ (cm<sup>-1</sup>): 1617, 789. MS m/z: 272 (M - 1). Anal. Found: C, 61.38; H, 9.74. C<sub>14</sub>H<sub>26</sub>Se Calc.: C, 61.52; H, 9.59%.

#### 3.1.5. cis-1-Methylseleno-1-cyclohexyl-1-octene (1c)

Oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>,  $\delta$  ppm): 6.21 (t, 1H, J = 7Hz); 2.40 (s, 3H); 1.90–2.30 (m, 3H); 0.63–1.75 (m, 21H). IR  $\nu$ (cm<sup>-1</sup>): 1616, 791. MS m/z: 286 (M - 1). Anal. Found: C, 62.57; H, 9.63. C<sub>15</sub>H<sub>28</sub>Se Calc.: C, 62.69; H, 9.84%.

## 3.1.6. trans-1-Methylseleno-1-cyclohexyl-1-ociene (2c)

Oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>,  $\delta$  ppm): 6.83 (t, 1H, J = 7 Hz); 2.49 (s, 3H); 1.91–2.29 (m, 3H); 0.62–1.74 (m, 21H). IR  $\nu$ (cm<sup>-1</sup>): 1614, 788. MS m/z: 286 (M - 1). Anal. Found: C, 62.93; H, 9.41. C<sub>15</sub>H<sub>28</sub>Se Calc.: C, 62.69; H, 9.84%.

#### 3.1.7. cis-1-Ethylseleno-1-cyclohexyl-1-octene (1d)

Oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>,  $\delta$  ppm): 6.18 (t, 1H, J = 7Hz); 2.76 (q, 2H, J = 7.3 Hz); 1.90–2.28 (m, 3H); 0.61–1.84 (m, 24H). IR  $\nu$ (cm<sup>-1</sup>): 1613, 788. MS m/z: 299 (M - 1). Anal. Found: C, 63.79; H, 10.19. C<sub>16</sub>H<sub>30</sub>Se Calc.: C, 63.97; H, 10.08%.

Table 1

Entry	Internal alkylselenoacetylene	Disubstituted alkenyl selenide	Yield * (%)	cis : trans <sup>b</sup>
	CH <sub>3</sub> Se===n-C <sub>4</sub> H <sub>9</sub>	CH <sub>3</sub> Se n-C <sub>4</sub> H <sub>0</sub>	69	36:64
•	C2H3Se==n-C4H9	c-C <sub>6</sub> H <sub>11</sub> C <sub>2</sub> H <sub>3</sub> Se n-C <sub>4</sub> H <sub>9</sub>	72	39:61
2	$CH_3Se^{-\Xi^{-}}n - C_6H_{13}$	с-С <sub>6</sub> Н <sub>11</sub> СН <sub>3</sub> Se n-С <sub>6</sub> Н <sub>13</sub>	77	31 : 69
i	$C_2H_5Se^{-2-n-}C_4H_{13}$	c-C <sub>6</sub> H <sub>11</sub> EtSe n-C <sub>6</sub> H <sub>13</sub>	68	34:66
•	$CH_3Se^{-\Xi-}n\cdot C_8H_{17}$	CH <sub>3</sub> Se n-C <sub>8</sub> H <sub>17</sub>	61	29:71
•	$C_2H_5Se = n \cdot C_8H_{17}$	c·C <sub>6</sub> H <sub>11</sub> C <sub>2</sub> H <sub>5</sub> Se n-C <sub>8</sub> H <sub>17</sub>	65	30:70
2	$C_2H_5Se = CH_2OCH_3$	c-C <sub>6</sub> H <sub>11</sub> C <sub>2</sub> H <sub>5</sub> Se CH <sub>2</sub> OCH <sub>3</sub>	57	21 : 79
•	CH <sub>3</sub> Se-=-Ph	c·C <sub>6</sub> H <sub>11</sub> CH₂Se ₽h	73	26:74
I	C <sub>2</sub> H <sub>5</sub> Se==-Ph	$C_2H_5Se$ Ph	75	29:71

<sup>a</sup> Isolated yield.

<sup>b</sup> Isomer ratio calculated from <sup>1</sup>H NMR data.

3.1.8. trans-1-Ethylseleno-1-cyclohexyl-1-octene (2d)

Oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>,  $\delta$  ppm): 6.81 (t, 1H, J = 7Hz); 2.75 (q, 2H, J = 7.3 Hz); 1.89–2.28 (m, 3H); 0.61–1.85 (m, 24H). IR  $\nu$ (cm<sup>-1</sup>): 1611, 788. MS m/z: 299 (M - 1). Anal. Found: C, 64.14; H, 10.22. C<sub>16</sub>H<sub>30</sub>Se Calc.: C, 63.97; H, 10.08%.

#### 3.1.9. cis-1-Methylseleno-1-cyclohexyl-1-decene (1e)

Oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>,  $\delta$  ppm): 6.15 (t, 1H, J = 7 Hz); 2.49 (s, 3H); 1.88–2.30 (m, 3H); 0.58–1.77 (m, 25H). IR  $\nu$ (cm<sup>-1</sup>): 1611, 781. MS m/z: 313 (M - 1). Anal. Found: C, 64.78; H, 10.42. C<sub>17</sub>H<sub>32</sub>Se Calc.: C, 64.94; H, 10.27%.

3.1.10. trans 1-Methylseleno-1-cyclohexyl-1-decene (2e) Oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>,  $\delta$  ppm): 6.81 (t, 1H, J = 7 Hz); 2.45 (s, 3H); 1.89–2.29 (m, 3H); 0.57–1.78 (m, 25H). IR  $\nu$ (cm<sup>-1</sup>): 1608, 783. MS *m*/*z*: 313 (*M* – 1). Anal. Found: C, 65.15; H, 10.01. C<sub>17</sub>H<sub>32</sub>Se Calc.: C, 54.94; H, 10.27%.

#### 3.1.11. cis-1-Ethylseleno-1-cyclohexyl-1-decene (1f)

Oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>,  $\delta$  ppm): 6.14 (t, 1H, J = 7Hz); 2.73 (q, 2H, J = 7.3 Hz); 1.87–2.28 (m, 3H); 0.58–1.83 (m, 28H). IR  $\nu$ (cm<sup>-1</sup>): 1609, 780. MS m/z: 327 (M - 1). Anal. Found: C, 65.67; H, 10.14. C<sub>18</sub>H<sub>34</sub>Se Calc.: C, 65.82; H, 10.45%.

## 3.1.12. trans-1-Ethylseleno-1-cyclohexyl-1-decene (2f)

Oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>,  $\delta$  ppm): 6.79 (t, 1H, J = 7Hz); 2.73 (q, 2H, J = 7.4 Hz); 1.87–2.25 (m, 3H); 0.55–1.85 (m, 28H). IR  $\nu$ (cm<sup>-1</sup>): 1604, 781. MS  $m/\epsilon$ : 327 (M = 1). Anal. Found: C, 65.94; H, 10.31. C<sub>18</sub>H<sub>M</sub>Se Calc.: C, 65.82; H, 10.45%.

3.1.13, cis-1=Ethylseleno=1=cyclohexyl=3-methoxy=1-pro= pene (1g)

Oil. <sup>T</sup>H NMR (CDCl<sub>3</sub>,  $\delta$  ppm): 7.11 (t, 1H, J = 6.8Hz); 2.88 (q, 2H, J = 7.1 Hz); 4.4 (d, 2H, J = 6.8 Hz); 3.75 (s, 3H); 2.17 (br, 1H); 1.16–1.66 (m, 10H). IR  $\nu$ (cm<sup>-1</sup>): 1609, 775. MS m/z: 260 (M - 1). Anal. Found: C, 54.93; H, 8.66; O, 6.27. C<sub>12</sub>H<sub>22</sub>Se Calc.: C, 55.16; H, 8.50; O, 6.12%.

#### 3.1.14. trans-1-Ethylseleno-1-cyclohexyl-3-methoxy-1propene (2g)

Oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>,  $\delta$  ppm): 6.54 (t, 1H, J = 6.8 Hz); 2.87 (q, 2H, J = 7.1 Hz); 4.44 (d, 2H, J = 6.8 Hz); 3.70 (s, 3H); 2.14 (br, 1H); 1.18–1.69 (m, 10H). IR  $\nu$ (cm<sup>-1</sup>); 1604, 770. MS m/z; 260 (M - 1). Anal. Found: C, 55.22; H, 8.55; O, 6.34. C<sub>12</sub>H<sub>22</sub>Se Calc.: C, 55.16; H, 8.50; O, 6.12%.

### 3.1.15. cis-1-Methylseleno-1-cyclohexyl-1-phenylethylene (1h)

Oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>, δ ppm): 7.10–7.50 (m, 5H); 6.83 (s, 1H); 2.44 (s, 3H); 2.31 (br, 1H); 0.97–1.55 (m, 10H). IR  $\nu$ (cm<sup>-1</sup>): 1654, 1597, 1549, 811. Anal. Found: C, 64.42; H, 7.31. C<sub>15</sub>H<sub>20</sub>Se Calc.: C, 64.51; H, 7.22%.

#### 3.1.16. trans-1-Methylseleno-1-cyclohexyl-1-phenylethylene (**2h**)

Oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>,  $\delta$  ppm): 7.04–7.50 (m, 5H); 6.75 (s, 1H); 2.41 (s, 3H); 2.28 (br, 1H); 0.95–1.55 (m, 10H). IR  $\nu$ (cm<sup>-1</sup>): 1647, 1588, 1550, 814. Anal. Found: C, 64.37; H, 7.15. C<sub>15</sub>H<sub>20</sub>Se Calc.: C, 64.51; H, 7.22%.

# 3.1.17. cis-1-Ethylseleno-1-cyclohexyl-1-phenylethylene (1i)

Oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>,  $\delta$  ppm): 7.0–7.55 (m, 5H); 6.81 (s, 1H); 2.69 (q, 2H, J = 7.1 Hz); 2.34 (br, 1H); 1.85 (t, 3H, J = 7.1 Hz); 0.98–1.60 (m, 10H). IR  $\nu$ (cm<sup>-1</sup>): 1661, 1590, 1544, 805. Anal. Found: C, 65.77; H, 7.66. C<sub>16</sub>H<sub>22</sub>Se Calc.: C, 65.52; H, 7.56%.

#### 3.1.18. trans-1-Ethylseleno-1-cyclohexyl-1-phenylethylene (2i)

Oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>,  $\delta$  ppm): 7.20–7.67 (m, 5H); 6.87 (s, 1H); 2.71 (q, 2H, J = 7.1 Hz); 2.25 (br, 1H); 1.81 (t, 3H, J = 7.1 Hz); 1.05–1.63 (m, 10H). IR  $\nu$ (cm<sup>-1</sup>): 1663, 1600, 1550, 807. Anal. Found: C, 65.49; H, 7.74. C<sub>16</sub>H<sub>22</sub>Se Calc.: C, 65.52; H, 7.56%.

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- [11] Preparation of 3 or 4: a mixture of 1a or 2a (1 mmol) and LiAlH<sub>4</sub> (1.5 mmol) in THF (5 ml) was heated at reflux for 3 h. After cooling to room temperature, methanol (1.5 mmol) was added to the mixture. The resulting mixture was stirred for 20 min and filtered. The filtrate was washed with saturated aq. NH<sub>4</sub>Cl (2 ml), separated, dried (MgSO<sub>4</sub>) and concentrated in vacuum to give a crude product which was purified by flash column chromatography. Eluting with pentane afforded 3 or 4. The reaction product contained pure *trans* 3 or *cis* 4, being consistent with the *trans* or *cis* isomers of known 1-cyclohexyl-
- 1-hexene. *trans* Isomer 3. <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta$  5.38 (multiplet, 2H); 0.8–2.3 (multiplet, 20H). <sup>13</sup>C NMR (CDCl<sub>3</sub>):  $\delta$  136.58; 127.81; 33.53; 32.55; 32.12; 26.35; 22.34; 14.03 ppm. IR (neat): 2880; 1645; 1450; 970 cm<sup>-1</sup>. GLC analysis, 98% pure. *cis* Isomer 4. <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta$  5.42 (multiplet, 2H); 0.78–2.2 (multiplet, 20H). <sup>13</sup>C NMR (CDCl<sub>3</sub>):  $\delta$  165.67; 126.83; 32.53; 31.79; 31.58; 26.17; 22.29; 14.01 ppm. IR (neat): 3019; 2876; 1640; 1448; 609 cm<sup>-1</sup>. GLC analysis, 98% pure.
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