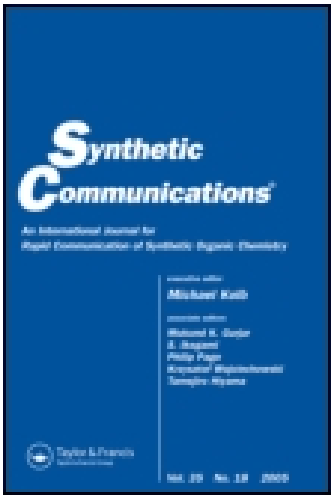


Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/lsyc20

Electrophilic Azido Selenenylation of Alkenes. A Simple Synthetic Route to Racemic Taxol Side Chain

Marcello Tiecco [a], Lorenzo Testaferri [a], Andrea Temperini [a], Luana Bagnoli [a], Francesca Marini [a] & Claudio Santi [a]
[a] Istituto di Chimica Organica, Università di Perugia, 06100, Perugia, Italy
Published online: 02 Sep 2006.

To cite this article: Marcello Tiecco , Lorenzo Testaferri , Andrea Temperini , Luana Bagnoli , Francesca Marini & Claudio Santi (1998) Electrophilic Azido Selenenylation of Alkenes. A Simple Synthetic Route to Racemic Taxol Side Chain, Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry, 28:12, 2167-2179, DOI: 10.1080/00397919808007031

To link to this article: http://dx.doi.org/10.1080/00397919808007031

SYNTHETIC COMMUNICATIONS, 28(12), 2167-2179 (1998)

ELECTROPHILIC AZIDO SELENENYLATION OF ALKENES. A SIMPLE SYNTHETIC ROUTE TO RACEMIC TAXOL SIDE CHAIN

Marcello Tiecco,* Lorenzo Testaferri, Andrea Temperini, Luana Bagnoli, Francesca Marini and Claudio Santi

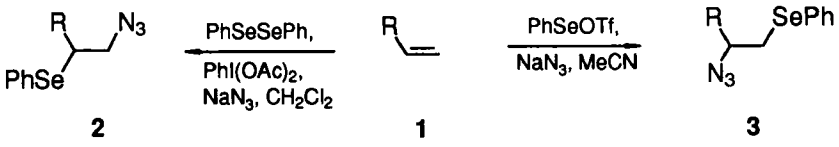
Istituto di Chimica Organica, Università di Perugia, 06100-Perugia, Italy

Abstract. Simple alkenes react with PhSeOTf and NaN_3 in MeCN to afford β-phenylseleno azides as the result of a stereospecific *trans* addition. The regioselectivity of the process is determined by the structure of the alkene.

We have recently reported that alkenes **1** easily react with PhSeSePh, NaN_3 and $PhI(OAc)_2$ to afford β-phenylseleno azides **2** (Scheme 1) as the result of a radical addition initiated by the azido radical.[1] Compounds **2** are useful intermediates which can find several synthetic applications.[2-7] For synthetic purposes it would be desirable to dispose of the regioisomeric compounds **3** which can be suggested to form from the electrophilic azido selenenylation of the same alkenes **1**. It has been reported that β-phenylseleno azides can be produced from the reaction of alkenes with PhSeCl and NaN_3 in DMSO.[8] Under these conditions, the addition reaction is stereospecific but it is not regiospecific. Similarly, the reaction of exocyclic alkenes

* To whom correspondence should be addressed

Scheme 1

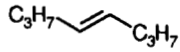
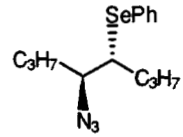
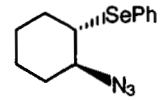
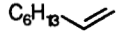
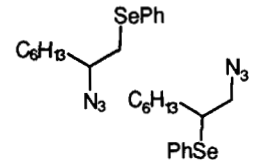
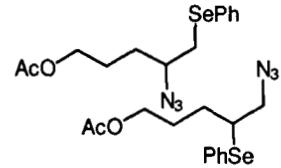
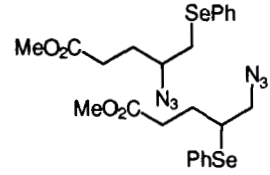
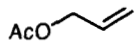
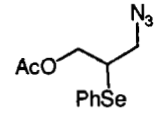
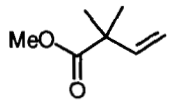
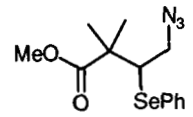


with N-(phenylseleno)phthalimide and azidotrimethylsilane in dichloromethane gives rise to a mixture of regioisomers.[9] We now report that β-phenylseleno azides can be easily obtained from the reaction of alkenes with phenylselenenyl triflate and NaN_3 in acetonitrile. This reaction is a stereospecific *trans* addition and occurs with a regiochemistry which is determined by the structure of the starting alkene (Scheme 1).

A first series of experiments was carried out at 25 °C with the alkenes indicated in Table 1. The reactions of (*E*)-4-octene and cyclohexene (entries 1 and 2) gave rise to the β-phenylseleno azides **4a** and **4b**, respectively, indicating that the reaction is a stereospecific *trans* addition. The regiochemical course of the addition was investigated using terminal alkenes (Table 1, entries 3-7). In the cases of 1-octene, 4-pentenyl acetate and methyl 4-pentenoate the reaction was not regiospecific and a mixture of regioisomers, **4c** and **4c'** (2:1), **4d** and **4d'** (1:1) and **4e** and **4e'** (2:1), was obtained. Only in the latter case the two isomers could be isolated by column chromatography. These results are similar to those reported for the reactions carried out in DMSO.[8] Interestingly, the reactions of allyl acetate and of methyl 2,2-dimethyl-3-pentenoate were regiospecific and afforded, although in low yield, the products **4f** and **4g** (Table 1, entries 6 and 7).

The formation of the anti-Markovnikov products is not completely unexpected. It has been observed that olefins containing an oxygen substituent in the allylic or

Table 1. Reactions of Alkenes with PhSeOTf and NaN_3 in MeCN at 25 °C.

Entry	Alkene	Time (h)	Phenylseleno Azide		Yield (%)
1		5		**4a**	60
2		4		**4b**	72
3		4		**4c** **4c'**	52 *a,b*
4		16		**4d** **4d'**	43 *a,b*
5		8		**4e** **4e'**	42 *a*
6		24		**4f**	40
7		15		**4g**	40

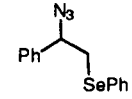
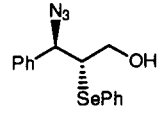
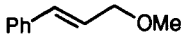
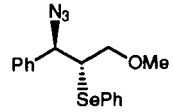
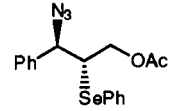
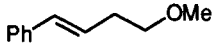
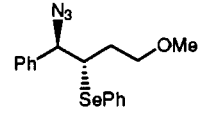
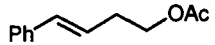
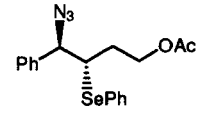
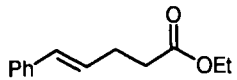
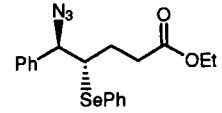
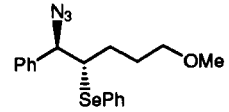
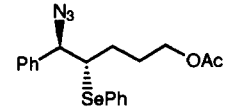
a) Mixture of regioisomers. *b*) Not separated.

homoallylic positions show a marked preference for anti-Markovnikov addition probably due to an attrattive interation between the selenium and the oxygen atoms in the seleniranium intermediate.[10,11]

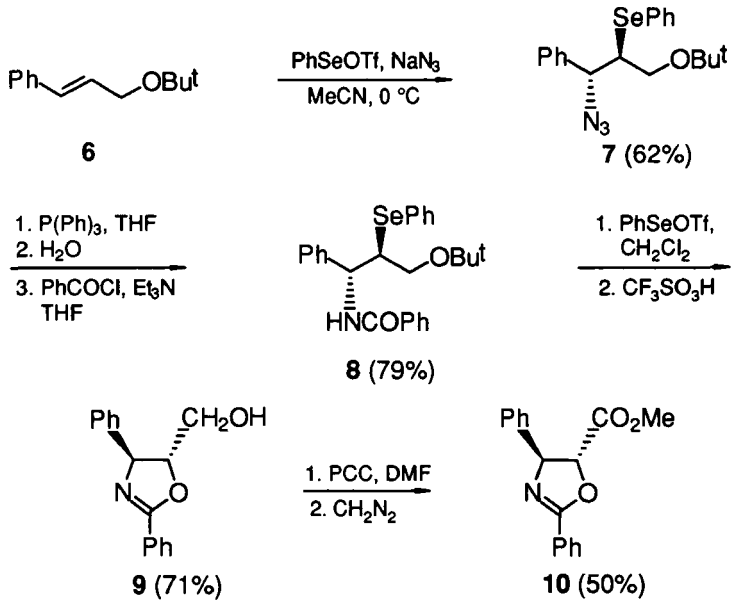
Complete regio and stereoselectivity was observed in the azido selenenylation of phenyl substituted alkenes (Table 2). These reactions were carried out at 0 °C. In fact, at 25 °C the reactions of styrene and of allylic derivatives (entries 1-4) gave rise to mixtures of products. The reactions of the other substrates (entries 5-9) at 25 °C gave instead results identical to those observed at 0 °C. As indicated in Table 2, the products **5b-5i** are the result of a stereospecific *trans* addition. The observed regioselectivity can be attributed to the presence of the phenyl group. In these cases the oxygen substituents do not seem to play a very important role since the same regioselectivity is observed when they occupy the allylic (entries 2-4), the homoallylic (entries 5, 6) or a more remote position (entries 7-9). The reaction of styrene gave the Markovnikov addition product **5a**. This result is interesting since the reaction carried out with PhSeCl in DMSO[8] afforded a mixture of the two regioisomers.

The azido selenenylation of alkenes described above has been employed to effect a simple synthesis of the oxazoline **10** (Scheme 2) which is known[12] to give the taxol side chain in two steps. The reaction of the *tert*-butyl cinnamyl ether **6** with PhSeOTf and NaN_3 in acetonitrile at 0 °C gave the phenylseleno azide **7** in 62% yield. This was converted into the benzamido derivative **8** by treatment with triphenylphosphine and then with benzoyl chloride. Treatment with PhSeOTf, according to our recently proposed procedure,[7] gave rise to the corresponding oxazoline as the result of an intramolecular nucleophilic deselenenylation reaction.

Table 2. Reactions of Phenyl Substituted Alkenes with PhSeOTf and NaN_3 in MeCN at 0 °C.

Entry	Alkene	Time (h)	Phenylseleno Azide		Yield (%)
1		3		**5a**	70
2		3		**5b**	60
3		1		**5c**	70
4		1		**5d**	70
5		2		**5e**	80
6		6		**5f**	74
7		2		**5g**	75
8		3		**5h**	65
9		2		**5i**	65

Scheme 2



Direct treatment with acid afforded the alcohol **9**. Oxidation with pyridinium chlorochromate gave the carboxylic acid which was directly treated with diazomethane to give the methyl ester **10**. Obviously, the oxazoline **10** produced in this way is the racemic compound. However, the procedure here described is extremely simple and very likely it can find practical applications.

Experimental

Starting materials were commercially available or were prepared by standard procedures. All new compound were characterized by MS, ^{1}H and ^{13}C NMR spectroscopy. GLC analyses and MS spectra were carried out with an HP 5890 gaschromatograph (dimethyl silicone column, 12.5 m) equipped with an HP 5971 Mass Selective Detector. ^{1}H and ^{13}C NMR spectra were recorded at 200 and

50.32 MHz, respectively, on a Bruker AC 200 instrument; $CDCl_3$ was used as solvent and TMS as standard. Elemental analyses were carried out on a Carlo Erba 1106 Elemental Analyzer.

Electrophilic azido selenenylation. General Procedure. To a solution of PhSeOTf,[13] prepared from PhSeCl (1.1 mmol) and AgOTf (1.1 mmol) in anhydrous CH_3CN (10 mL) under N_2, was added NaN_3 (4.0 mmol) and then the substrate (1.0 mmol) dissolved in anhydrous CH_3CN. The mixture was stirred at 25 °C or at 0 °C (see text) and the progress of the reaction was monitored by TLC and GC-MS. The reaction mixture was filtered through anhydrous K_2CO_3 and the filtrate was evaporated under vacuum.

The residue was chromatographed through a silica gel column using mixtures of petroleum ether and ether (from 100/0 to 30/70) as eluants. Reaction products, reaction times and yields are indicated in Tables 1 and 2. Compounds **4b**,[8] **4c**,[14] **4c'**,[1] **4e'**,[2] **4g**[2] and **5a**,[1] have already been described. Compound **5b**, after acetylation, was identical to **5d**. Physical and spectral data of the other reaction products are reported below.

(4*SR*,5*RS*)-4-Azido-5-(phenylseleno)octane (4a):[1] oil; ^1H NMR δ 7.65-7.40 (m, 2 H), 7.43-7.18 (m, 3 H), 3.50 (dt, 1 H, J = 6.5, 4.6 Hz), 3.24-3.12 (m, 1 H), 1.85-1.20 (m, 8 H), 0.92 (t, 3 H, J = 7.1 Hz), 0.88 (t, 3 H, J = 7.0 Hz); ^{13}C NMR δ 134.8, 131.4, 129.1, 127.6, 66.6, 51.5, 34.6, 33.3, 21.3, 19.8, 13.8, 13.7. MS m/z (relative intensity) 311 (14), 213 (21), 170 (53), 157 (65), 126 (78), 77 (34), 55 (100), 41 (74).

4-Azido-5-(phenylseleno)pentyl acetate (4d) and **5-azido-4-(phenylseleno) pentyl acetate (4d')**: (1:1 mixture) oil; ^{1}H NMR δ 7.62-7.48 (m, 4 H), 7.35-7.22 (m, 6 H), 4.14-3.98 (m, 4 H), 3.64-3.42 (m, 3 H), 3.26-3.10 (m, 1 H), 3.10-2.97 (m, 2 H), 2.05 (s, 3 H), 2.02 (s, 3 H), 2.00-1.50 (m, 8 H); ^{13}C NMR δ 170.8, 170.2, 135.3, 133.1, 129.1, 128.1, 127.4, 63.6, 62.2, 55.7, 43.9, 32.3, 30.7, 28.8, 26.7, 25.0, 20.7. Anal. Calcd for $C_{13}H_{17}N_3O_2Se$: C, 47.86; H, 5.25; N, 12.88. Found: C, 47.75, H, 5.33, N, 13.00.

Methyl 4-azido-5-(phenylseleno)pentanoate (4e): oil; ^{1}H NMR δ 7.62-7.49 (m, 2 H), 7.36-7.24 (m, 3 H), 3.66 (s, 3 H), 3.64-3.40 (m, 1 H), 3.10-3.00 (m, 2 H), 2.70-2.38 (m, 2 H), 2.14-2.00 (m, 1 H), 1.90-1.70 (m, 1 H); ^{13}C NMR δ 173.0, 135.4, 129.2, 128.0, 61.7, 51.6, 32.3, 30.4, 29.4. Anal. Calcd for $C_{12}H_{15}N_3O_2Se$: C, 46.16; H, 5.25; N, 12.88. Found: C, 46.21; H, 5.13; N, 12.80.

3-Azido-2-(phenylseleno)propyl acetate (4f): oil; ^{1}H NMR δ 7.65-7.50 (m, 2 H), 7.34-7.21 (m, 3 H), 4.39 (dd, 1 H, J = 11.6, 5.2 Hz), 4.25 (dd, 1 H, J = 11.6, 7.3 Hz), 3.68-3.58 (m, 2 H), 3.48-3.32 (m, 1 H), 2.05 (s, 3 H); ^{13}C NMR δ 170.2, 135.1, 129.2, 128.3, 126.9, 64.3, 52.6, 41.6, 20.5. MS m/z (relative intensity) 299 (18), 157 (52), 130 (32), 77 (43), 43 (100). Anal. Calcd for $C_{11}H_{13}N_3O_2Se$: C, 44.31; H, 4.39; N, 14.09 Found: C, 44.43; H, 4.36; N, 14.14.

(1*RS*,2*RS*)-1-Azido-3-methoxy-1-phenyl-2-(phenylseleno)propane (5c): oil; ^{1}H NMR δ 7.42-7.33 (m, 2 H), 7.30-7.24 (m, 5 H), 7.24-7.09 (m, 3 H), 4.96 (d, 1 H, J = 6.6 Hz), 3.83-3.70 (m, 1 H), 3.59-3.46 (m, 2 H), 3.34 (s, 3 H); ^{13}C NMR δ 136.9, 134.9, 128.8, 128.3, 127.6, 72.4, 66.4, 58.7, 50.2. MS m/z (relative intensity) 347 (20), 215 (26), 183 (100), 157 (64), 130 (27), 117 (18), 104 (30),

91 (24), 77 (63), 45 (32). Anal. Calcd for $C_{16}H_{17}N_3OSe$: C, 55.51; H, 4.95; N, 12.13. Found: C, 55.48; H, 4.86; N, 12.20.

(2*RS*,3*RS*)-3-Azido-3-phenyl-2-(phenylseleno)propyl acetate (5d): oil; ^1H NMR δ 7.48-7.38 (m, 2 H), 7.36-7.12 (m, 8 H), 4.89 (d, 1 H, J = 6.8 Hz), 4.45 (dd, 1 H, J = 11.8, 5.5 Hz), 4.28 (dd, 1 H, J = 11.8, 6.3 Hz), 3.60 (ddd, 1 H, J = 6.9, 6.3, 5.5 Hz), 2.00 (s, 3 H); ^{13}C NMR δ 170.2, 136.6, 134.8, 128.9, 128.5, 127.9, 127.2, 66.8, 64.0, 48.3, 20.4. MS m/z (relative intensity) 375 (2), 201 (21), 183 (20), 157 (15), 104 (22), 77 (34), 43 (100). Anal. Calcd for $C_{17}H_{17}N_3O_2Se$: C, 54.55; H, 4.58; N, 11.23. Found: C, 54.41; H, 4.50; N, 11.16.

(1*RS*,2*SR*)-1-Azido-4-methoxy-1-phenyl-2-(phenylseleno)butane (5e): oil; ^1H NMR δ 7.54-7.40 (m, 2 H), 7.37-7.20 (m, 8 H), 4.79 (d, 1 H, J = 5.8 Hz), 3.68-3.42 (m, 3 H), 3.18 (s, 3 H), 2.20-2.00 (m, 1 H), 1.84-1.60 (m, 1 H); ^{13}C NMR δ 137.8, 134.7, 129.0, 128.4, 128.1, 127.6, 127.2, 70.2, 70.0, 58.2, 48.7, 30.2. MS m/z (relative intensity) 361 (9), 229 (30), 198 (35), 117 (30), 91 (30), 71 (29), 45 (100). Anal. Calcd for $C_{17}H_{19}N_3OSe$: C, 56.67; H, 5.31; N, 11.66. Found: C, 56.60; H, 5.38; N, 11.73.

(3*SR*,4*RS*)-4-Azido-4-phenyl-3-(phenylseleno)butyl acetate (5f): oil; ^1H NMR δ 7.53-7.41 (m, 2 H), 7.39-7.26 (m, 8 H), 4.78 (d, 1 H, J = 6.0 Hz), 4.34-4.15 (m, 2 H), 3.41 (ddd, 1 H, J = 9.8, 6.0, 3.5 Hz), 2.24-2.02 (m, 1 H), 1.96-1.70 (m, 1 H), 1.90 (s, 3 H); ^{13}C NMR δ 170.6, 137.6, 135.1, 129.1, 128.3, 128.0, 127.1, 69.9, 62.5, 48.6, 29.6, 20.7. MS m/z (relative intensity) 389 (7), 195 (62), 157 (25), 144 (59), 130 (41), 116 (100), 104 (49), 91 (22), 77 (39), 43 (49). Anal.

Calcd for $C_{18}H_{19}N_3O_2Se$: C, 55.67; H, 4.93; N, 10.82. Found: C, 55.61; H, 4.99; N, 10.88.

Ethyl (4*SR*,5RS)-5-azido-5-phenyl-4-(phenylseleno)pentanoate (5g): oil; ^1H NMR δ 7.50-7.38 (m, 2 H), 7.38-7.12 (m, 8 H), 4.75 (d, 1 H, J = 6.2 Hz), 4.05 (q, 2 H, J = 7.1 Hz), 3.38 (ddd, 1 H, J = 10.3, 6.2, 3.4 Hz), 2.74-2.40 (m, 2 H), 2.26-2.05 (m, 1 H), 1.94-1.70 (m, 1 H), 1.18 (t, 3 H, J = 7.1 Hz); ^{13}C NMR δ 172.0, 137.3, 134.6, 128.6, 128.0, 127.8, 127.4, 126.8, 69.6, 59.7, 51.2, 31.9, 25.5, 13.7. MS m/z (relative intensity) 375 (12), 218 (17), 191 (47), 157 (23), 144 (74), 130 (100), 117 (94), 104 (49), 77 (60). Anal. Calcd for $C_{19}H_{21}N_3O_2Se$: C, 56.73; H, 5.26; N, 10.45. Found: C, 56.81; H, 5.13; N, 10.61.

(1*RS*,2*SR*)-1-Azido-5-methoxy-1-phenyl-2-(phenylseleno)pentane (5h): oil; ^1H NMR δ 7.52-7.40 (m, 2 H), 7.40-7.12 (m, 8 H), 4.71 (d, 1 H, J = 6.2 Hz), 3.48-3.20 (m, 3 H), 3.26 (s, 3 H), 2.05-1.50 (m, 4 H); ^{13}C NMR δ 137.8, 134.9, 128.9, 128.3, 128.0, 127.6, 127.1, 72.0, 69.7, 58.2, 52.0, 27.7, 27.2. Anal. Calcd for $C_{18}H_{21}N_3OSe$: C, 57.75; H, 5.65; N, 11.23. Found: C, 57.70; H, 5.72; N, 11.21.

(4*SR*,5*RS*)-5-Azido-5-phenyl-4-(phenylseleno)pentyl acetate (5i): oil; ^1H NMR δ 7.52-7.42 (m, 2 H), 7.37-7.14 (m, 8 H), 4.72 (d, 1 H, J = 6.1 Hz), 3.99 (t, 2 H, J = 6.3 Hz), 3.39-3.27 (m, 1 H), 2.10-1.52 (m, 4 H), 1.95 (s, 3 H); ^{13}C NMR δ 170.2, 137.7, 135.0, 128.9, 128.3, 128.1, 127.7, 127.0, 69.6, 63.5, 51.3, 26.6, 26.4, 20.6. Anal. Calcd for $C_{19}H_{21}N_3O_2Se$: C, 56.72; H, 5.26; N, 10.44. Found: C, 56.79; H, 5.17; N, 10.39.

(1*RS*,2*RS*)-1-Azido-3-(*tert*-butoxy)-1-phenyl-2-(phenylseleno)propane (7): oil; ^1H NMR δ 7.38-7.02 (m, 10 H), 5.00 (d, 1 H, J = 7.0 Hz), 3.83 (dd, 1 H, J = 8.7,

3.5 Hz), 3.60-3.40 (m, 2 H), 1.16 (s, 9 H); ^{13}C NMR δ 137.2, 134.6, 128.7, 128.2, 127.8, 127.3, 73.4, 66.7, 62.2, 51.3, 27.4. MS m/z (relative intensity) 389 (5), 200 (33), 184 (30), 157 (22), 134 (36), 117 (33), 104 (45), 77 (48), 57 (100). Anal. Calcd for $C_{19}H_{23}N_3OSe$: C, 58.76; H, 5.97; N, 10.82; Found: C, 58.81; H, 5.85; N, 10.93.

1-[(1*RS*,2*RS*)-3-(*tert*-Butoxy)-1-(phenylcarboxamido)-2-(phenylseleno)propyl] benzene (8): The phenylseleno azide **7** (1.0 mmol) and Ph_3P (1.1 mmol) were dissolved in anhydrous THF (20 mL) and were stirred at 40 °C overnight. Then H_2O (50.0 mmol) was added and the mixture was stirred at 50 °C for 3 h. The solution was diluted with C_6H_6 and evaporated. The residue was further dried over P_2O_5 under vacuum for 5 h, then it was dissolved in anhydrous THF (20 mL) at 0 °C, and Et_3N (2.0 mmol) and PhCOCl (1.1 mmol) were added. After 3 h the reaction mixture was poured on water and extracted with CH_2Cl_2. The organic layer was washed with brine, dried and evaporated. The crude product was chromatographed on silica gel using petroleum ether and ether (60/40) as eluant. The product **8** was obtained as oil: ^1H NMR δ 8.49 (d, 1 H, J = 7.4 Hz), 8.00-7.88 (m, 2 H), 7.72-7.60 (m, 2 H), 7.54-7.47 (m, 3 H), 7.34-7.14 (m, 8 H), 5.54 (dd, 1 H, J = 7.4, 3.9 Hz), 3.72-3.46 (m, 3 H), 1.22 (s, 9 H); ^{13}C NMR δ 166.6, 140.3, 135.0, 134.7, 131.2, 129.1, 128.3, 127.9, 127.1, 127.0, 126.2, 74.1, 62.1, 57.2, 50.2, 27.3. Anal. Calcd for $C_{26}H_{29}NO_2Se$: C, 66.94; H, 6.27; N, 3.00. Found: C, 66.87; H, 6.18; N, 2.95.

[(4*SR*,5*RS*)-2,4-Diphenyl-4,5-dihydro-1,3-oxazol-5-yl]methanol (9): To a solution of PhSeOTf (1.1 mmol) in CH_2Cl_2 (10 mL) the selenoacetamide **8** (1.0

mmol) was added at room temperature. The progress of the reaction was monitored by TLC. The cyclized product was then treated with CF_3SO_3H to remove the *tert*-butyl ether function and to give alcohol **9**. After 1 h the reaction mixture was neutralyzed with NH_4OH 30% and extracted with CH_2Cl_2. The organic layer was dried (Na_2SO_4) and evaporated. The crude product was chromatographed on silica gel using petroleum ether and ether (60/20) as eluant. The alcohol **9** was obtained as oil: ^1H NMR δ 8.08-7.95 (m, 2 H), 7.55-7.19 (m, 8 H), 5.04 (d, 1 H, J = 7.4 Hz), 4.49 (ddd, 1 H, J = 7.4, 5.4, 3.8 Hz), 3.80 (dd, 1 H, J = 12.2, 3.8 Hz), 3.68 (dd, 1 H, J = 12.2, 5.4 Hz), 3.3 (br s, 1 H); ^{13}C NMR δ 164.2, 141.7, 131.6, 128.7, 128.4, 128.3, 127.6, 127.2, 126.6, 87.7, 71.4, 63.2. MS m/z (relative intensity) 253 (10), 193 (100), 105 (20), 89 (22). Anal. Calcd for $C_{16}H_{15}NO_2Se$: C, 57.84; H, 4.55; N, 4.22. Found: C, 57.90; H, 4.61; N, 4.17.

Methyl [(4*SR*,5*RS*)-2,4-diphenyl-4,5-dihydro-1,3-oxazole]-5-carboxylate (10): The dihydrooxazole **9** (1.0 mmol) and PCC (3.5 mmol) were dissolved in anhydrous DMF (20 mL) and stirred for 24 h at room temperature. Then excess CH_2N_2 was added at 0 °C. The reaction mixture was poured on water and was extracted with ether. The organic layer was washed with brine, dried (Na_2SO_4) and evaporated. The residue was chromatographed on silica gel using petroleum ether and ether (60/40) as eluant to give pure ester **10**, whose spectroscopic data are identical to those reported.[12]

Acknowledgements. Financial support from the CNR, Rome, and Ministero dell'Università e della Ricerca Scientifica e Tecnologica, Italy, is gratefully acknowledged.

References

1. Tingoli, M.; Tiecco, M.; Chianelli, D.; Balducci, D.; Temperini, A. *J. Org. Chem.*, **1991**, *56*, 6809.
2. Tingoli, M.; Tiecco, M.; Testaferri, L.; Andrenacci, R.; Balducci, D. *J. Org. Chem.*, **1993**, *58*, 6097.
3. Santoyo-González, F.; Calvo-Flores, F. G.; García-Mendoza, P.; Hernández-Mateo, F.; Isac-García, J.; Robles-Díaz, R. *J. Org. Chem.*, **1993**, *58*, 6122.
4. Czernecki, S.; Randriamandimby, D. *Tetrahedron Lett.*, **1993**, *34*, 7915.
5. Czernecki, S.; Ayadi, E.; Randriamandimby, D. *J. Chem. Soc., Chem. Commun.*, **1994**, 35.
6. Tingoli, M.; Tiecco, M.; Testaferri, L.; Temperini, A. *J. Chem. Soc., Chem. Commun.*, **1994**, 1883.
7. Tingoli, M.; Testaferri, L.; Temperini, A.; Tiecco, M.; *J. Org. Chem.*, **1996,** *61*, 7085.
8. Hassner, A.; Amarasekara, A. S. *Tetrahedron Lett.*, **1987**, *28*, 5185.
9. Giuliano, R. M.; Duarte, F. *Synlett*, **1991**, 419.
10. Engman, L. *J. Org. Chem.*, **1989**, *54*, 884.
11. Cooper, M. A.; Ward, A. D. *Tetrahedron Lett.*, **1995**, *36*, 2327.
12. Gou, D. M.; Liu, Y. C.; Chen, C. S. *J. Org. Chem.*, **1993**, *58*, 1287; Chen, S.-H.; Farina, V.; Vyas, D. M.; Doyle, T. W.; Long, B. H.; Fairchild, C. *J. Org. Chem.*, **1996**, *61*, 2065.
13. Murata, S.; Suzuki, T. *Chem. Lett.*, **1987**, 849.
14. Denis, J. N.; Vicens, J.; Krief, A. *Tetrahedron Lett.*, **1979**, 2697.

(Received in the UK 19 November 1997)