The Reaction of Wittig Reagents with Selenium. Formation of Selenoaldehydes

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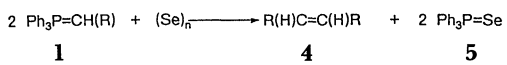
The reaction of Wittig reagents with elemental selenium gave the corresponding selenoaldehydes which further reacted with other Wittig reagents to give the corresponding dimeric olefins in good yields. The selenoaldehydes formed afforded corresponding adducts by the reaction with dienes. These selenoaldehydes obtained by retro Diels-Alder reaction were also found to react with Wittig reagents to give the corresponding olefins in good yields.

Phosphonium ylides [**1**] have been widely utilized for the synthesis of olefins and also known to react with sulfur to afford thiocarbonyl compounds.[1,2] Recently, we found that the reaction of stable Wittig reagents (**1**) with episulfides or elemental sulfur afforded dialkyl fumarates or maleates in good yields and confirmed that these reactions proceeded through thioaldehyde intermediates.[3] This consideration, in turn, prompted us to investigate the possibility of the formation of selenocarbonyl compounds by the reaction of elemental selenium with **1**.

Selenoaldehydes [**2**] are interest compounds for their anomalous reactivity.[4] Until our preliminary communication in 1987, however, there had been no report on the formation of **2** from **1**.[5] At almost the same time with our communication, Erker, Hock, and Nolte reported the generation of **2** by the reaction of **1** with elemental selenium.[6] In this paper, we report the reaction of **1** with elemental selenium.

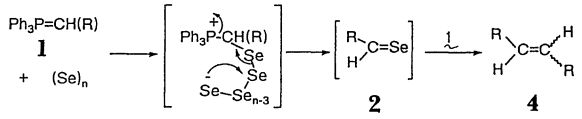
Preparation of Olefins. Compounds **1** were prepared by the reaction of corresponding phosphonium salts [**3**] with bases and isolated in the case of stable reagents. We first tried the reaction of stable **1** with elemental selenium. Treatment of (methoxycarbonylmethylene)triphenylphosphorane [**1a**] with elemental selenium afforded dimethyl fumarate [**4a**] and triphenylphosphine selenide [**5**] in 74% and 81% yields, respectively (Scheme 1). Other reactions were carried out in a similar manner. When the stable ylides **1** were used as substrates, only trans olefins [**4**] were isolated in all instances. Erker et al. also reported that this reaction proceeded by use of catalytic amount of selenium or **5**.[6] We also tried the reaction of **1a** with 1/5 equiv of **5**. However, no reaction occurred by treatment of this ylide with **5**. The reactivity between stable ylides and alkyl-substituted ylides seems to be quite different.



Scheme 1.

In a previous paper, we also reported the reaction of **1** with elemental sulfur, which afforded the same dimeric olefins.[3] The present reaction seems to proceed in a similar manner via a selenoaldehyde intermediate. The ylide carbanion attacks elemental selenium to give the corresponding **2**, which further reacts with **1** to afford **4** as shown in Scheme 2.

Generation of Selenoaldehydes. Many workers confirmed the formation of **2** by trapping it with dienes.[4] Then, we tried cycloaddition reactions to trap selenoaldehyde intermediates. Treatment of (phenylmethylene)triphenylphosphorane [**1c**] with 2,3-dimethyl-1,3-butadiene in the presence of elemental selenium in refluxing toluene afforded the corresponding Diels-Alder adduct [**6c**], **4c**, and **5** in 34%,



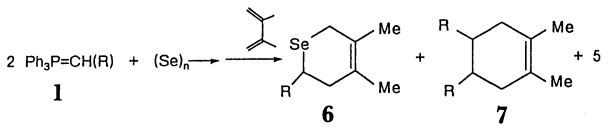
Scheme 2.

Table 1. Reaction of Phosphonium Ylides with Elemental Selenium

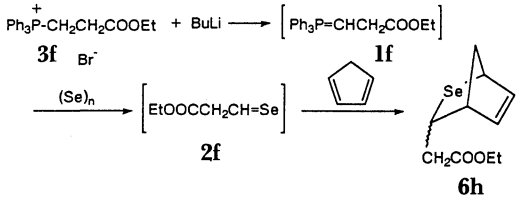
1	Compound R	Conditions				Products		Yields/% trans/cis[a]
		Solvent	Temperature/°C	Time/h		4	5	
a	COOMe	toluene	reflux	4	4a	74	81	trans only
b	COOEt	toluene	reflux	4	4b	91	95	trans only
c	Ph	toluene	reflux	2	4c	68	79	4
c	Ph	THF	reflux	1	4c	65	85	8
c	Ph	THF	rt	2	4c	41	67	10
c	Ph	toluene	0	8	4c	51	63	10
d	CN	toluene	reflux	4	4d	45	54	trans only
e	COMe	toluene	reflux	4	4e	46	71	trans only

a) The ratio was determined by their NMR spectra.

54%, and 76% yields, respectively. Interestingly, when **1a** was used as a substrate, compound [**7a**] was obtained in 16% yield along with **6a** (62%) and **5** (65%) (Scheme 3). Compound **7a** is the reaction product of **4a** with 2,3-dimethylbuta-1,3-diene. As shown in Table 2, Diels-Alder adducts were obtained in moderate yields. Olefin **4a** is the by-product of the present reaction. 2,3-Dimethyl-1,3-butadiene might be less reactive than stable **1a**. Then, we chose cyclopentadiene as a substrate, which is more reactive than



Scheme 3.



Scheme 4.

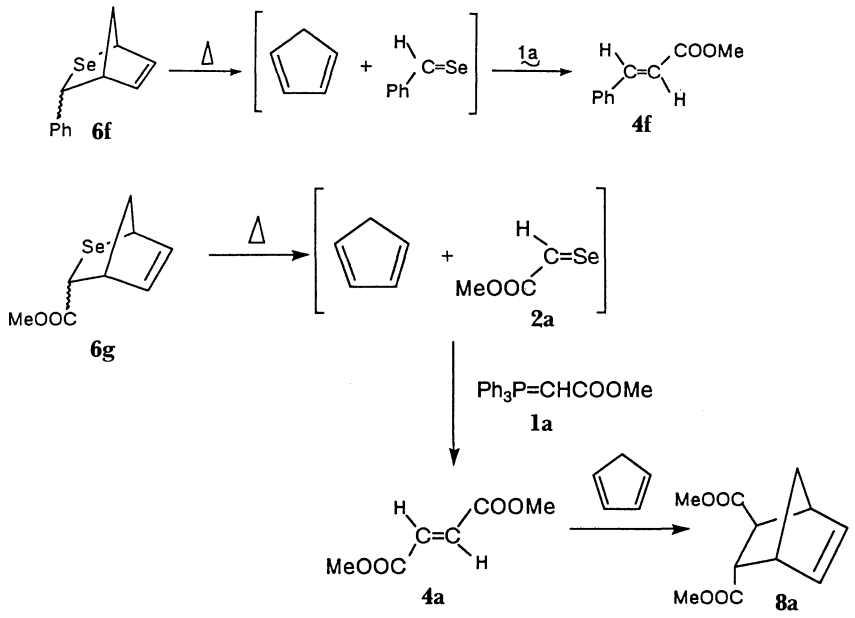
dimethylbutadiene. When 2 equiv of Se was added to a solution of ylide **1a** (or **1c**), followed by the addition of cyclopentadiene (5 equiv), the desired adduct **6f** (or **6g**) was obtained in 27% (or 25%) yield. In the past decade, many workers reported the new synthetic methods for **2**: Reid et al. prepared stable heterocyclic selenoaldehydes by Vilsmeier reaction;[4a] reaction of α-silyl selenocyanates with Bu_4NF;[4b] 1,2-elimination of selenosulfates;[4c] reaction of aldehyde with bis(trimethylsilyl)selenide in the presence of catalytic BuLi.[4d] Recently, Okazaki and coworkers reported the isolation of **2**.[4e] However, there is no report on the preparation of **2** from phosphonium ylides except our and Elker's communications.[5,6] The present method has many advantages: the reaction simply occurred by mixing two reagents (ylides and selenium). Selenoaldehydes containing a variety of functionalities can be easily prepared. For example, the Diels-Alder adduct, **6h**, was prepared by two step process from triphenylphosphine and ethyl 2-bromopropionate as shown in Scheme 4.

Reaction of Selenoaldehydes with Wittig Reagents by a Retro-Diels-Alder Reaction. Recently, Kirby et al. and Elker et al. reported that anthracene (or cyclopentadiene)-selenobenzaldehyde adduct was

Table 2. Reaction of Selenoaldehydes with Diene

Selenoaldehyde R	Conditions Temperature/°C	Se/equiv	Diene/equiv		Products Yield/% 4	5	6	7
COOMe	90	3.3	5	**4a**	0	65	**6a** 62	**7a** 16
COOEt	reflux	3.3	5	**4b**	0	85	**6b** 42	**7b** 6
Ph	90	3.3	5	**4c**	54	76	**6c** 34	**7c** 0
$COCH_3$	reflux	2	5	**4d**	0	40	**6d** 14	**7d** 7[a]
CN	reflux	2	5	**4e**	0	40	**6e** 13	**7e** 4[a]

a) Compounds **7d** and **7e** could not be isolated in pure form.



Scheme 5.

decomposed to selenobenzaldehyde and anthracene (or cyclopentadiene) upon heating at 75 °C, which further reacted with 2,3-dimethyl-1,3-butadiene to afford another Diels-Alder adduct.[4c,6] This result is another preparative method for **2**. So, we tried the reaction **6f** or **6g** with **1** in toluene (Scheme 5). Treatment of ylide **1a** with the adduct [**6f**] in refluxing toluene afforded methyl cinnamate (**4f**) and **5** in 95% and 85% yields, respectively. We also tried the reaction of **1a** with **6g** in refluxing toluene. In this case, another Diels-Alder adduct [**8a**] was obtained in 68% yield along with **4a** and **5**. This adduct is the reaction product of cyclopentadiene with **4a**. It is interesting that cyclopentadiene formed by thermolysis further reacted with **4a** to give **8a**. Then we tried this reaction in the presence of cyclopentadiene (3 equiv). Compound **8a** was obtained in 88% yield. As shown in Table 3, the corresponding olefins were obtained in good yields. The reaction might proceed through selenaphosphetane intermediates. However, Vedejs and co-workers reported that the reaction of thiopivaldehyde with (3-phenylpropylidene)triphenylphosphorane afforded the corresponding episulfide in 32% yield.[7] If this reaction is applicable to selenoaldehydes, the reaction of **1** with **2** would have afforded olefins via episelenide intermediates, which are known as unstable compounds. At present, we do not have any evidence which route is operative.

Table 3. Reaction of Diels-Alder Adducts with 2,3-Dimethyl-1,3-butadiene

Adduct	Ylide	Product Yield/%			
		Olefin	4	8	5
6f R=Ph	**1a**	**4f**	95	**8f** 0	85
	1b	**4g**	94	**8g** 0	94
6g R=COOMe	**1a**	**4a**	32	**8a** 67	87
	1b	**4h**	24	**8h** 62	97
	1c	**4f**	0	**8f** 0	23

We also found that the reaction of adduct (**6a**) with 2,3-dimethyl-1,3-butadiene in refluxing toluene afforded another adduct in 64% yield.

In summary, the reaction of **1** with elemental selenium gave the dimeric compounds **4** in good yields. This reaction proceeds through selenoaldehyde intermediates. The experimental results in this paper indicate that selenoaldehydes containing a variety of functionalities can be prepared easily and participate with good efficiency in cycloaddition reactions. The retro-Diels-Alder reaction made another approach for the generation of selenoaldehydes, which further reacted with phosphonium ylides to afford olefins in good yields.

Experimental

General Methods. Melting points are uncorrected. NMR spectra were obtained by using JEOL PMX-60, FX-90Q, and GSX 400 spectrometers. Mass spectra were recorded on a JEOL GS 270 spectrometer.

Materials. Stable phosphonium ylides were prepared by a method mentioned in the literature.[1] Cyclopentadiene was obtained from dicyclopentadiene by distillation. 2,3-Dimethyl-1,3-butadiene was purchased from Aldrich Chem. and used without further purification. Authentic dimethyl fumarate, diethyl fumarate, fumaric acid, methyl cinnamate, and ethyl cinnamate were purchased from Aldrich Chem.

General Procedure for Synthesis of Olefins 4 from phosphonium Ylides 1. To a solution of containing 3.3 g (10 mmol) of carbomethoxymethylenetriphenylphosphorane (**1a**) in toluene (100 ml) was added a suspension of selenium (0.79 g, 10 mmol) in 15 ml of toluene. After refluxing for 4 h, the reaction mixture was evaporated and the remaining oil was extracted three times with hexane. The combined extracts were evaporated and roughly chromatographed on silica gel to give dimethyl fumarate (**4a**) (0.53 g, 3.7 mmol) in 74% yield. Other reactions were carried out in a similar manner.

Reaction of 1 with Catalytic Amount of 5. To a solution of **1a** (1.0 g, 3.0 mmol) in 30 ml of toluene was added compound **5** (0.26 g, 0.75 mmol) in one portion. After refluxing for 24 h, the solution was evaporated to give colorless crystals, which was recrystallized from methanol to give colorless crystals of **1a** (0.80 g, 2.4 mmol, 80%).

Cycloadduct (6b) of 2,3-Dimethylbuta-1,3-diene and Ethyl Selenoxoacetate Derived from Ylide 1b. To a solution of (ethoxycarbonylmethylene)triphenylphosphorane (**1b**) (1.00 g, 3.0 mmol) and 2,3-dimethylbuta-1,3-diene (4.11 g, 50 mmol) in toluene (50 ml) was added elemental selenium (0.79 g, 10 mmol) in one portion. After stirring for 20 h at 90 °C, the reaction mixture was cooled, filtered, and evaporated to give pale orange oily crystals. This residue was extracted with pentane (5 ml) for three times. The combined extracts were evaporated and distilled to afford a mixture of adducts **6b** and **7b** (130—140 °C/2 mmHg, 1 mmHg=133.322 Pa). This oil was chromatographed over silica gel by elution with hexane-dichloromethane (4 : 1) to give **6b**[4c] (0.31 g, 1.3 mmol, 42%) and **7b** (0.02 g, 0.08 mmol, 6%). Compound **7b** was identical with the authentic sample. Other reactions were carried out in a similar manner.

Preparation of Authentic Diels-Alder Adducts (7b). To a solution of diethyl fumarate (1.2 g, 8.3 mmol) in toluene was added a solution of 2,3-dimethylbuta-1,3-diene in toluene (20 ml). After refluxing for 20 h, the reaction mixture was evaporated to give a pale yellow oil, which was distilled under reduced pressure (bulb-to-bulb distillation) to give colorles oil of **7b**. (130—140 °C/2 mmHg, 1.25 g, 4.9 mmol, 59%), ^{1}H NMR ($CDCl_3$) δ=1.25 (t, CH_3, J=7.3 Hz, 6H), 1.62 (s, CH_3, 6H), 2.17 (m, 2H), 2.25—2.29 (m, 2H), 2.79—2.82 (m, 2H), 4.14 (q, CH_2, J=7.3 Hz and J=3.1 Hz, 4H). Precise mass for $C_{14}H_{22}O_4$: 254.1518 (calcd), 254.1490 (found). Other reactions were carried out in a similar manner. **7a** 110—120 °C/2 mmHg, bulb-to-bulb distillation); ^{1}H NMR ($CDCl_3$) δ=1.62 (s, CH_3, 6H), 2.16 (m, 2H), 2.25—2.29 (m, 2H), 2.82—2.84 (m, 2H), 3.69 (s, OCH_3 6H). **7d** (120—130 °C/2 mmHg); ^{1}H NMR ($CDCl_3$) δ=1.57 (s, CH_3, 6H), 1.79—1.88 (m, 2H), 2.14 (s, 6H, COMe), 2.12—2.22 (m, 2H), 2.85—2.95 (m, 2H).

Reaction of 1a with Senium in the Presence of Cyclopentadiene. To a solution containing 1 mmol of ylide (**1a**) (0.33 g) and cyclopentadiene (0.33 g, 5 mmol) in toluene (30

ml) was added elemental selenium (0.16 g, 2.0 mmol) at 90 °C. After stirring for 14 h, the resulting mixture was evaporated to give brown oily crystals. The resulting mixture was extracted with hexane (30 ml) for three times. The combined extracts were evaporated to afford a pale orange oil, which was distilled to give the cycloadducts [**6g**] as a mixture of endo and exo isomers. **6g**;[4c] (80—90 °C/1.2 mmHg, 0.059 g, 0.27 mmol, 27%); ^{1}H NMR ($CDCl_3$) (Exo isomer) δ=1.86 (d, 1H, J=10.2 Hz), 2.05 (d, 1H, J=9.5 Hz), 3.29 (m, 1H), 3.54 (m, 1H), 3.73 (s, CH_3), 4.43 (m, 1H), 5.75—5.77 (m, =CH), 6.40—6.42 (m, =CH). (Endo isomer) δ=1.65—1.68 (m, 1H), 1.78—1.80 (m, 1H), 3.46 (m, 1H), 3.72 (s, CH_3), 4.37 (m, 1H), 4.69 (d, 1H, J=3.6 Hz), 5.91—5.92 (m, =CH), 6.46—6.48 (m, =CH). Compound **6f** was obtained in a similar manner. **6f**;[4d] (100—110 °C/1.2 mmHg, 0.059 g, 0.25 mmol, 25%); ^{1}H NMR ($CDCl_3$) (Endo isomer) δ=1.86 (br s, 2H, CH_2), 3.33 (br, 2H), 4.45 (br, 1H), 5.24 (d, CH, J=3.9 Hz), 5.51—5.53 (m, =CH), 6.55—6.57 (m, =CH), 7.17—7.48 (m, 5H). (Exo isomer) ($CDCl_3$) δ=1.88—1.89 (m, 1H), 2.12—2.16 (m, 1H), 3.11 (br, 1H), 4.32 (s, 1H), 4.55 (br, 1H), 5.94—5.96 (m, =CH), 6.44—6.46 (m, =CH) 7.15—7.58 (m, 5H, Ar).

Reaction of Phosphonium Ylide 1f with Selenium and Cyclopentadiene. To a suspension of [2-(ethoxycarbonyl)-ethyl]triphenylphosphonium bromide[7] (2.22 g, 5.0 mmol) in toluene (50 ml) was added a solution of butyllithium (4.5 ml, 10% w/v, 7.0 mmol) in hexane at room temperature. After stirring for 1 h, cyclopentadiene (11.7 g, 25 mmol) and elemental selenium (0.79 g, 10 mmol) were added in portionwise to this orange suspension which was then refluxed for 6 h. The resulting suspension was filtered off and evaporated to give dark brown oily crystals, which were extracted with pentane (20 ml×3). The residue was recrystallized from benzene to give colorless crystals of **5** (0.74 g, 2.2 mmol, 44%). The combined extracts were evaporated and distilled under reduced pressure (bulb-to-bulb distillation, 100—110 °C/1.5 mmHg) to give a colorless oil of **6h** (0.36 g, 1.46 mmol, 29%). Compound **6h** was obtained as a mixture of isomers. ^{1}H NMR ($CDCl_3$) δ=1.23 (t, CH_3), 1.27 (t, CH_3), 1.33—1.54 (m, 6H). 1.86—1.91 (m, 2H), 2.16 (m, 1H), 2.90—2.94 (m, 3H), 3.02 (s, 1H), 3.19 (s, 1H), 4.06 (q, 2H), 4.13 (q, 2H), 5.90—5.92 (m, 1H), 6.10—6.12 (m, 2H), 6.16—6.18 (m, 1H). Precise mass for $C_{10}H_{14}O_2Se$ (^{80}Se): 246.0159 (calcd), 246.0087 (found).

The Reaction of Diels-Alder Adduct 6g with Phosphonium Ylide 1a. To a solution of **6g** (0.109 g, 0.50 mmol) in toluene (15 ml) was added a solution of **1a** (0.167 g, 0.50 mmol) in toluene (10 ml). After refluxing for 38 h, the reaction mixture was evaporated to give brown oily crystals, which were extracted with pentane (10 ml×3). The combined extracts were evaporated to give an orange oil, which was distilled under reduced pressure (bulb-to-bulb distillation). Dimethyl fumarate (**4a**) was first distilled (60—70 °C/1.2 mmHg, 0.023 g, 0.16 mmol, 32%). Compound **8a** was lately distilled (100—110 °C/1.2 mmHg, 0.071 g, 0.034 mmol, 68%). ^{1}H NMR ($CDCl_3$) δ=1.44—1.47 (m, 1H), 1.61 (d, 1H, J=8.8 Hz), 2.66 (d, 1H, J=2.7), 3.11 (m, 1H), 3.25 (m, 1H), 3.35 (m, 1H), 3.64 (s, Me), 3.71 (s, Me), 6.06 (m, 1H), 6.26 (m, 1H). Compound **8a** was hydrolyzed in 5 M NaOH to give the corresponding dicarboxylic acid. Mp 170—171 °C (lit.[9] 166—168 °C); ^{1}H NMR (D_2O, outernal TMS) δ=1.48—1.62 (m, CH_2), 2.63 (m, 1H), 3.16—3.50 (m, 3H), 6.12 (m, 1H), 6.29 (m, 1H). Other reactions were carried out in a similar manner. **4h**; (ethyl methyl fumarate); bp (60—70 °C/1.2 mmHg, 0.019 g, 0.12 mmol, 24%). ^{1}H NMR ($CDCl_3$) δ=1.32 (t, CH_3, J=7.3 Hz), 3.81 (s, CH_3), 4.26 (q, CH_2, J=7.3 and 14.0 Hz), 6.86 (s, 2H). ^{13}C NMR ($CDCl_3$) δ=14.11, 52.29, 61.36, 133.13, 133.94, 164.93, 165.48. This mixed ester was hydrolyzed in 5 M NaOH to give the corresponding fumaric acid; mp 297—299 °C. **8h** was obtained as a 1 : 1 mixture of 1α,2α,3β,4α and 1α,2β,3α,4α adducts. **8h**; 100—110 °C/1.2 mmHg, 0.069 g, 0.31 mmol, 62%. ^{1}H NMR ($CDCl_3$) δ=1.24 (t, CH_3, J=7.3 Hz), 1.45 (d, 1H, J=9.1 Hz), 1.61 (d, 1H, J=9.1 Hz), 2.66—2.67 (m, 1H), 3.10 (m, 1H), 3.25 (m, 1H), 3.37 (t, 1H, J=4.2 Hz), 3.71 (s, CH_3), 4.09 (q, J=7.0 and 7.3 Hz), 6.05—6.06 (m, =CH), 6.26—6.27 (m, =CH). Another isomer 1.29 (t, CH_3, J=7.3 Hz), 1.45 (d, 1H, J=9.1 Hz), 1.61 (d, 1H, J=9.1 Hz), 2.64 (m, 1H), 3.10 (m, 1H), 3.25 (m, 1H), 3.34 (t, J=4.27 Hz), 3.64 (s, CH_3), 4.16 (d, J=6.71 and 7.33 Hz), 6.05—6.06 (m, =CH), 6.26—6.27 (m, =CH). This mixture was hydrolyzed in 5 M NaOH to give the corresponding dicarboxylic acid; mp 170—171 °C (lit.[9] mp 166—168 °C).

Reaction of Diels-Alder Adduct 6f with Ylide 1a. To a solution of adduct **6f** (0.12 g, 0.50 mmol) in toluene (10 ml) was added a solution of ylide **1a** (0.17 g, 0.50 mmol) in toluene (5 ml). After refluxing for 38 h, the resulting solution was evaporated and extracted with hexane (10 ml×3). The combined extracts were evaporated to give a pale yellow oil, which was distilled under reduced pressure (bulb-to-bulb distillation, 90—100 °C/1.2 mmHg). Methyl cinnamate (**4f**) (trans only) was obtained in 95% yield. (0.077 g, 0.48 mmol) The reaction of **6f** with ylide **1b** was carried out in a similar manner; ethyl cinnamate (**4g**) (trans only) (**4g**); bp 90—100 °C/1.2 mmHg (0.083 g, 0.47 mmol, 94%).

Reaction of 6g with 1a Followed by the Addition of Cyclopentadiene. To a solution of **6g** (0.046 g, 0.30 mmol) in toluene (15 ml) was added a solution of **1a** (0.10 g, 0.30 mmol). After refluxing for 6 h, a solution of cyclpentadiene (0.13 g, 2.0 mmol) in toluene (5 ml) was added to this solution. After refluxing for 16 h, the solution was evaporated to give pale brown oily crystals. The resulting mixture was extracted with pentane (10 ml×3). The combined extracts were evaporated to give a pale yellow oil, which was distilled under reduced pressure (bulb-to-bulb distillation). The colorless oil of **8a** was obtained (100—110 °C/1.5 mmHg, 0.050 g, 0.26 mmol, 88%).

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