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Facile and Efficient Way to Synthesize the Radical Cyclization Precursor Methyl 3-(tert-Butyl((E)-3-(2,2-diphenylcyclopropyl)-2-p for Kinetic Research

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Facile and Efficient Way to Synthesize the Radical Cyclization Precursor Methyl 3-(*tert*-Butyl((*E*)-3-(2,2-diphenylcyclopropyl)-2-propenyl)amino)-3-Oxo-2-(phenylseleno)propanoate for Kinetic Research

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Abstract: Methyl 3-(*tert*-butyl((*E*)-3-(2,2-diphenylcyclopropyl)-2-propenyl)amino)-3-oxo-2-(phenylseleno)propanoate was prepared in 10 steps in good to excellent yield using benzophenone and hydrazine hydrate as the starting materials.

Keywords: carbene reaction, IBX, phenylselenization, selective reduction, Wittig reaction

Knowledge of the rate constants of radical reactions is very important because most useful radical reactions involve chain processes with several competing pathways available. One can adjust the appropriate reagent concentrations in advance of experimentation with the knowledge so that the desired sequence will be efficient. Consequently, research on the rate constants of radical reactions has received a great deal of attention.[1] Newcomb developed a general method for direct laser flash photolysis (LFP) measurements of unimolecular radical kinetics with ultraviolet (UV) – detectable diphenyl radical reporter groups [Eq. (1)].[2a] They have used LFP to examine the radical

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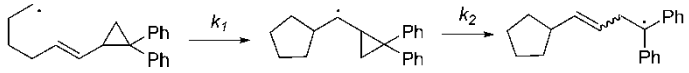
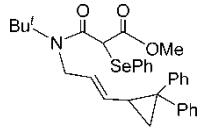
rearrangement reactions of a number of radicals of phenylselenide derivatives and pyridinethioneoxycarbonyl (PTOC) ester derivatives.[2]



(1)

However, the radicals they examined were limited to all-carbon skeletons. The radical cyclization percursors containing heteroatoms such as amide ester have not been studied. Considering that the introduction of a heteroatom will bring different electronic effects to the whole molecule and further affect the reaction rate of the radical cyclization, a backbone containing a nitrogen atom with a diphenyl radical reporter group needs to be synthesized to study the kinetic character of the radical cyclization. Here we report a facile and efficient way to a cyclization precursor: methyl 3-(*tert*-butyl((*E*)-3-(2,2-diphenylcyclopropyl)-2-propenyl)amino)-3-oxo- 2-(phenylselanyl)propanoate **(1)**.

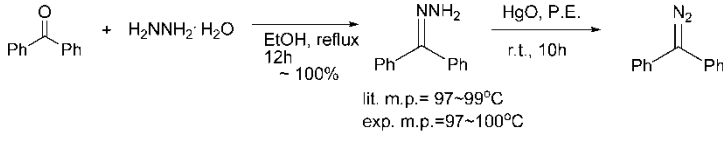


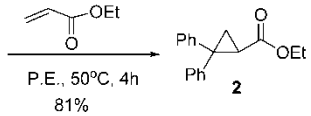
1

The coupling of the secondary amine **7** and potassium monomethyl malonate results in the formation of methyl 3-(*tert*-butyl((*E*)-3-(2,2-diphenylcyclopropyl)-2-propenyl)amino)-3-oxopropanoate, which could be converted into the target compound **1** by phenylselenization. The secondary amine **7** could be generated by the method developed by Frøyen and Juvvik.[3] The double bond and the three-member ring could be constructed by modified Wittig reaction and carbene reaction, respectively (Scheme 1).

There are two methods to construct the three-membered ring by the carbene reaction.

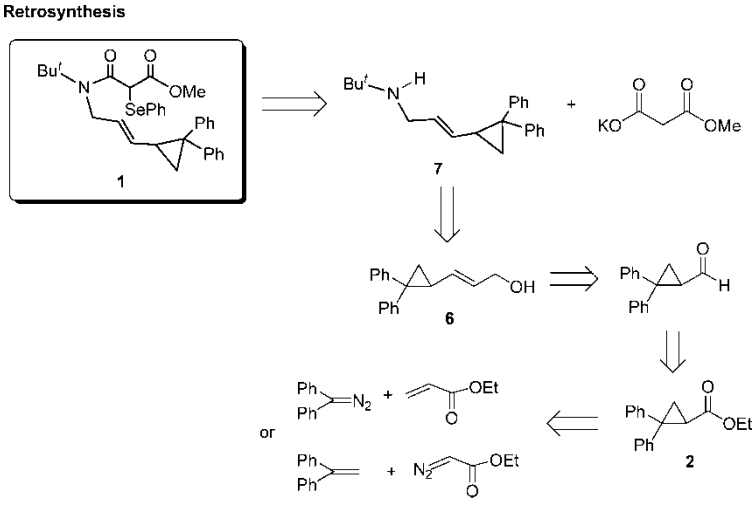
METHOD A





2

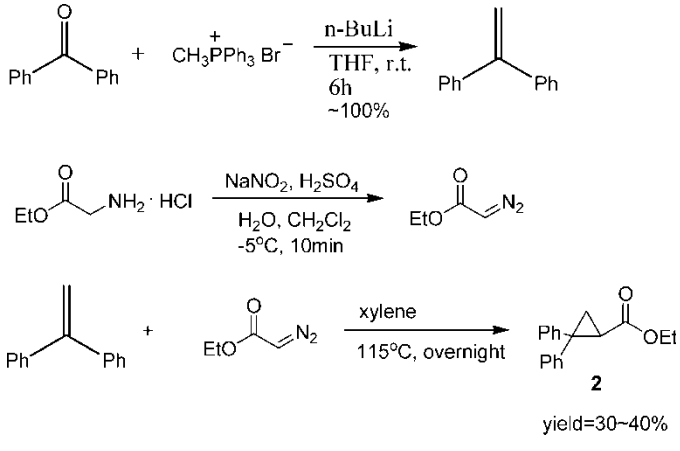
Benzophenone was refluxed with 86% hydrazine hydrate in ethanol to



Scheme 1. Retrosynthetic analysis of the compound **1**.

quantitatively yield the corresponding hydrazone. The benzophenone hydrazone was treated with yellow oxide of mercury,[4] and after filtration, the purple petroleum ether solution of diphenyldiazomethane was used directly in the next step. The carbene reaction occurred with the characteristic phenomenon of decoloration and nitrogen evolution.[5] The yield was good.

METHOD B

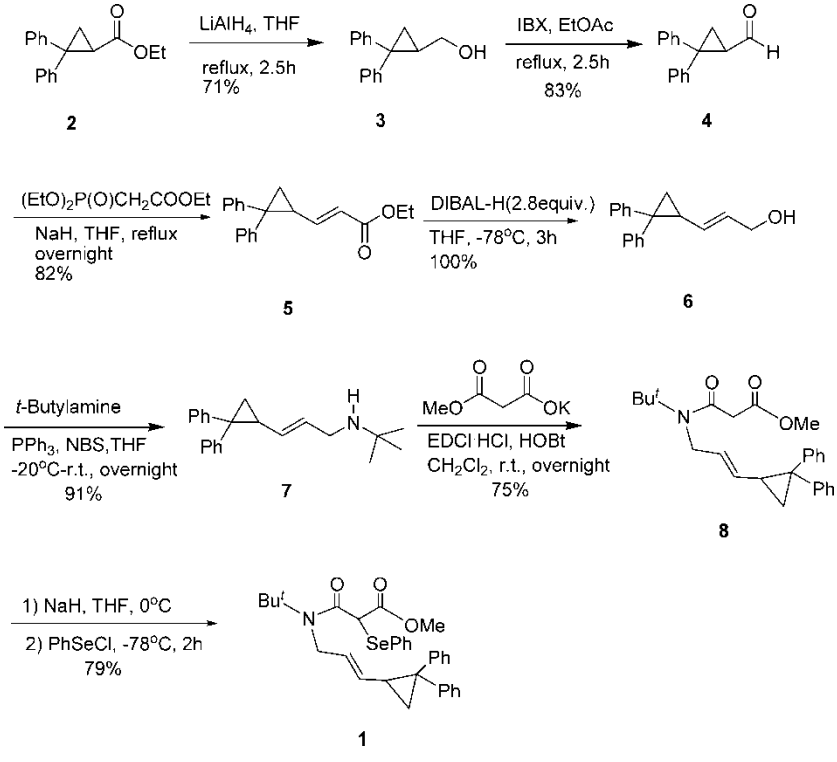


1,1-Diphenylethene was quantitatively prepared by Wittig reaction from benzophenone. Ethyl glycinate hydrochloride was diazotized to ethyl

diazoacetate.[6] The carbene reaction took place at high temperature, but the result was not satisfactory.[7]

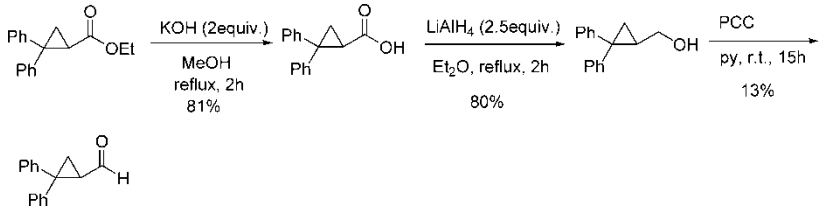
Comparing these two methods, we found each has its advantage. Method A's, advantages are 1) diphenyldiazomethane is relative stable and safe, 2) the large excess of ethyl acrylate (bp = 99°C) can be removed on a rotary evaporator, 3) the thin-layer chromatography (TLC) is clear. Its disadvantage is that in the preparation of diphenyldiazomethane, a by-product is toxic mercury and workup must be careful. Method B's advantage is that no toxic substance is produced. Disadvantages are 1) ethyl diazoacetate is very active and dangerous, and in the preparation, concentration of the solvent must be carried out carefully; 2) recovery of the large excess of 1,1-diphenylethene by column costs great vigor; and 3) the TLC is messy. Purification is difficult. Method **A** therefore is superior to method **B**.

The synthetic sequence for the generation of compound **1** is depicted in Scheme 2. In the literature, the alcohol **3** was obtained from ethyl 2,2-diphenylcyclopropanecarboxylate **(2)** via hydrolysis with potassium hydroxide followed by reduction of the resulting acid with lithium aluminium



Scheme 2.

hydride (LAH) in dry ether in good yield. However, the yield in the oxidation was low and the environmentally unfriendly oxidative reagent pyridinium chlorochromate (PCC) was used.[8]



We directly reduced the ester to alcohol with $LiAlH_4$, skipping the hydrolysis step. The mild and economic oxidative reagent, IBX(*o*-iodoxybenzoic acid),[9] was used, and the yield was excellent. The construction of double bond was furnished by a Horner–Wadsworth–Emmons reaction with triethylphosphonoacetate and sodium hydride in THF in good yield, and the ester group was selectively reduced by DIBAL-H at $-78^{\circ}C$.[10] The $LiAlH_4$ is not suitable for this situation because it will partly reduce the double bond. The secondary amine was prepared by a one-pot synthesis from the alcohol **6** and *t*-butylamine via alkoxyphosphonium salt.[3] The coupling of the secondary amine **7** and potassium monomethyl malonate was carried out employing the couple reagent 1-[3-(dimethylamino)propyl]-3-ethylcarbodiimide hydrochloride (EDCI) and *N*-hydroxybenzotriazole (HOBt). EDCI can be neutralized with potassium salt so that the reaction proceeds smoothly. The amide ester **8** deprotoned by sodium hydride was treated with phenylselenyl chloride and finally gave the target compound **1**.[11]

In conclusion, we have elaborated a facile and efficient way to the radical cyclization precursor **1** for kinetic research. Compound **1** was obtained from commercially available reagents in 10 steps in good to excellent yield. The related kinetic measurement is in progress.

EXPERIMENTAL

Commercially available reagents were purchased from either the Sinopharm Group Shanghai Chemical Reagent Company or the Acros chemical companies and were used as received. Tetrahydrofuran (THF) and diethyl ether were distilled from sodium benzophenone ketyl. Methylene chloride was distilled from calcium hydride. ^1H NMR spectra (400 MHz) and ^{13}C NMR spectra (100 MHz) were obtained on EXA 400 spectrometers. Mass spectra were recorded with a Finnigan MAT 95 mass spectrometer for both low-resolution and high-resolution mass spectra.

Ethyl 2,2-Diphenylcyclopropanecarboxylate (2)

Benzophenone hydrazone (2.94 g, 15 mmol), yellow oxide of mercury (3.25 g, 15 mmol), and 15 ml of petroleum ether were placed in a bottle. The bottle was wrapped with aluminum foil and stirred slowly at rt for 10 h. The deep purple mixture was filtered to remove mercury and any benzophenone azine. The filtrate was used directly in the next step. A solution of diphenyldiazomethane in 15 ml of petroleum ether was added in portions, with stirring, at 50°C to ethyl acrylate (2.96 g, 4.3 ml, 39.54 mmol). Each portion decolorized gradually. The addtion time was about 4 h. The solvent was removed in vacuo, and the residue was purified by flash column chromatography to give **2** (3.23 g, 81%) as a light yellow oil. Analytical TLC (silica gel 60), 20% EtOAc in *n*-hexane, $R_f = 0.74$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36–7.14 (m, 10H), 3.96–3.86 (m, 2H), 2.54 (dd, $J = 6.0$, 7.8 Hz, 1H), 2.17 (t_{obs}, $J = 5.3$ Hz, 1H), 1.58 (dd, $J = 3.4$, 4.8 Hz, 1H), 1.01 (t, $J = 7.1$ Hz, 3H).

(2,2-Diphenylcyclopropyl)methanol (3)

A solution of **2** (1.646 g, 6.18 mmol) in THF (3 ml) was added dropwise to a stirred suspension of lithium aluminum hydride (494 mg, 12.36 mmol) in THF (30 ml). After refluxing for 2.5 h, water was carefully added. The mixture was transferred into a separatory funnel along with diethyl ether and 30 ml of 5% HCl soltuion. The layers were separated, and the aqueous layer was extracted with ether (3 × 30 ml). The combined organic layer was then dried over anhydrous Na_2SO_4. The drier was filtered, and the filtrate was concentrated. The crude product was purified by flash-column chromatography to give **3** (0.983 g, 71%) as a yellow oil. Analytical TLC (silica gel 60), 20% EtOAc in *n*-hexane, $R_f = 0.41$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39–7.12 (m, 10H), 3.50–3.46 (m, 1H), 3.39–3.35 (m, 1H), 2.04–1.98 (m, 1H), 1.40–1.37 (m, 1H), 1.31–1.26 (m, 2H, include -OH).

2,2-Diphenylcyclopropanecarbaldehyde (4)

Compound **3** (0.993 g, 4.43 mmol) was dissolved in EtOAc (30 ml), and *o*-iodoxybenzoic acid (IBX) was added. The resulting suspension was immersed in an oil bath set at 80°C and stirred vigorously open to the atmosphere. After 2 h, the reaction mixture was cooled to rt and filtered. The filtrate was concentrated, and the residue was purified by flash-column chromatography to give **4** (0.821 g, 83%) as a yellow oil. Analytical TLC (silica gel 60), 20% EtOAc in *n*-hexane, $R_f = 0.80$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.67 (d, $J = 6.9$ Hz, 1H), 7.42–7.17 (m, 10H), 2.58–2.52 (m, 1H), 2.26 (dd, $J = 5.3$, 5.3 Hz, 1H), 1.89 (dd, $J = 5.3$, 8.5 Hz, 1H).

Ethyl (*E*)-3-(2,2-diphenylcyclopropyl)acrylate (5)

Ethyl diethylphosphonoacetate (0.937 g, 0.83 ml, 4.18 mmol) was added dropwise to a suspension of NaH (184 mg, 60% mineral oil dispersion, 4.60 mmol) in THF (4 ml) at 0°C over 5 min, and the resulting mixture was stirring at rt for 30 min. A solution of **4** (0.844 g, 3.8 mmol) in THF (1 ml) was added to the mixture. The reaction mixture was stirred at reflux for 30 min and then at rt for 12 h. Aqueous NH_4Cl solution (1 N) was added to the mixture. The organic solvent was removed under reduced pressure. The aqueous layer was extracted by Et_2O (3 × 30 ml). The combined organic layer was then dried over anhydrous Na_2SO_4. The drier was filtered, and the filtrate was concentrated. The crude product was purified by flash-column chromatography to give **5** (0.907 g, 82%) as a yellow oil. Analytical TLC (silica gel 60), 20% EtOAc in *n*-hexane, $R_f = 0.66$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33–7.14 (m, 10H), 6.24 (dd, $J = 10.3$, 15.4 Hz, 1H), 5.97 (d, $J = 15.1$ Hz, 1H), 4.11 (q, $J = 7.1$ Hz, 2H), 2.39–2.36 (m, 1H), 1.80 (dd, $J = 5.0$, 8.7 Hz, 1H), 1.71 (t_{obs}, $J = 5.3$ Hz, 1H), 1.22 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.4, 150.2, 145.6, 140.5, 130.6, 128.7, 128.5, 127.4, 127.2, 126.4, 120.0, 60.1, 39.7, 30.2, 23.4, 14.3; IR (neat) 2981, 1714, 1643 cm^{-1}; LRMS for $C_{20}H_{20}O_2$ (EI, 20 eV) m/z 293 ($M^+ + 1$, 5), 292 (M^+, 31); HRMS (EI) for $C_{20}H_{20}O_2$ (M^+): calcd. 292.1463, found 292.1467.

(*E*)-3-(2,2-Diphenylcyclopropyl)prop-2-en-1-ol (6)

Compound **5** (0.665 g, 2.27 mmol) was added to a solution of DIBAL (1 M solution in toluene, 6.37 ml, 6.37 mmol) and THF (10 ml) at −78°C over 5 min, and the resulting mixture was stirred at −78°C for 3 h. MeOH (1 ml) was added to the mixture at −78°C, followed by water (1 ml). The mixture was allowed to warm to rt for 0.5 h. Water (1 ml) was added to the mixture, followed by 15 wt% aqueous NaOH solution (1 ml). The white precipitate was filtered, and the filtrate was concentrated in vacuo. The residue was diluted by Et_2O. The aqueous layer was extracted by Et_2O (3 × 30 ml). The combined organic layer was then dried over anhydrous Na_2SO_4. The drier was filtered, and the filtrate was concentrated. The crude product was purified by flash-column chromatography to give **6** (0.568 g, 100%) as a yellow oil. Analytical TLC (silica gel 60), 20% EtOAc in *n*-hexane, $R_f = 0.46$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39–7.11 (m, 10H), 5.79 (dt, $J = 6.2$, 15.6 Hz, 1H), 4.97 (dd, $J = 9.4$, 15.4 Hz, 1H), 3.96 (d, $J = 6.0$ Hz, 2H), 2.31–2.25 (m, 1H), 1.59 (dd, $J = 5.0$, 8.7 Hz, 1H), 1.49 (t_{obs}, $J = 5.3$ Hz, 1H), 1.24 (br, 1H, -OH); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.5, 141.3, 133.9, 130.9, 128.8, 128.4, 127.4, 126.7, 126.0, 63.7, 37.3, 29.6, 22.2; IR (neat) 2925, 1660, 1599 cm^{-1}; LRMS for $C_{18}H_{18}O$ (EI, 20 eV) m/z

250 (M^+, 9); HRMS (EI) for $C_{18}H_{18}O$ (M^+): calcd. 250.1358, found 250.1375.

(*E*)-*N*-*tert*-Butyl-3-(2,2-diphenylcyclopropyl)prop-2-en-1-amine (7)

N-Bromosuccimide (404 mg, 2.27 mmol) was added to a stirred solution of triphenylphosphine (595 mg, 2.27 mmol) and **6** (568 mg, 2.27 mmol) in anhydrous THF (10 ml) at −20°C over 2–3 min in small portions under N_2. After 5 min, *tert*-butylamine (498 mg, 0.72 ml, 6.81 mmol) was injected via a syringe in one portion. The temperature was raised to rt naturally overnight. Petroleum ether (10 ml) was added to the reaction mixture and stirred for 0.5 h. The phosphine oxide and succinimide solid were precipitated. The solid was filtered and washed by HCl (1 N). Then the aqueous layer was neutralized by $NaHCO_3$ solution and extracted by Et_2O (3 × 10 ml). The combined organic layer was then dried over anhydrous Na_2SO_4. Filtered and concentrated. The crude product was purified by flash-column chromatography to give **7** (632 mg, 91%) as a light yellow oil. Analytical TLC (silica gel 60), 50% EtOAc in *n*-hexane, $R_f = 0.17$; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.34–7.10 (m, 10H), 5.79 (dt, $J = 6.9$, 15.1 Hz, 1H), 4.97 (dd, $J = 9.2$, 15.4 Hz, 1H), 3.03 (ABdd, $J_{AB} = 12.1$ Hz, $J = 0.9$, 6.8 Hz, 2H), 2.25 (dt, $J = 5.9$, 8.7 Hz, 1H), 1.55 (dd, $J = 5.0$, 8.7 Hz, 1H), 1.47 (t_{obs}, $J = 5.3$ Hz, 1H), 1.04 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.7, 141.5, 132.5, 131.1, 129.3, 128.34, 128.28, 127.4, 126.5, 125.9, 50.4, 45.0, 37.0, 29.7, 29.1, 22.0; IR (neat) 2962, 1599, 1495 cm^{-1}; LRMS for $C_{22}H_{27}N$ (EI, 20 eV) m/z 305 (M^+, 12); HRMS (EI) for $C_{22}H_{27}N$ (M^+): calcd. 305.2143, found 305.2172.

Methyl 3-(*tert*-butyl((*E*)-3-(2,2-diphenylcyclopropyl)-2-propenyl)amino)-3-oxopropanoate (8)

Compound **7** (0.632 g, 2.07 mmol), potassium monomethylmalonate (0.388 g, 2.48 mmol), EDCI HCl (0.619 g, 3.23 mmol), and HOBt (0.503 g, 3.73 mmol) were dissolved in 50 ml CH_2Cl_2 at 0°C and stirred at rt overnight. The white precipitate was filtered, and the organic layer was washed with saturated $NaHCO_3$ solution (30 ml) and hydrochloric acid (1 N) (30 ml). It was then dried over anhydrous Na_2SO_4. The drier was filtered, and the filtrate was concentrated. The crude product was purified by flash-column chromatography to give **8** (0.631 g, 75%) as a light yellow oil. Analytical TLC (silica gel 60), 50% EtOAc in *n*-hexane, $R_f = 0.43$; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32–7.12 (m, 10H), 5.55 (dt, $J = 4.6$, 15.6 Hz, 1H), 4.89 (dd, $J = 8.9$, 15.3 Hz, 1H), 3.85–3.69 (m, 2H), 3.72 (s, 3H), 3.25 (AB, $J_{AB} = 15.1$ Hz, 2H), 2.32 (dt, $J = 6.0$, 8.7 Hz, 1H), 1.58–1.52 (m, 2H), 1.26 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.9, 167.1, 146.3, 141.1, 132.8, 130.8, 128.6, 128.4,

127.4, 126.8, 126.7, 126.1, 57.8, 52.4, 47.1, 43.6, 37.3, 29.4, 28.6, 21.9; IR (neat) 2958, 1746, 1651 cm^{-1}; LRMS for $C_{26}H_{31}NO_3$ (EI, 20 eV) m/z 406 ($M^+ + 1$, 19), 405 (M^+, 42); HRMS (EI) for $C_{26}H_{31}NO_3$ (M^+): calcd. 405.2304, found 405.2325.

Methyl 3-(*tert*-butyl((*E*)3-(2,2-diphenylcyclopropyl)-2-propenyl)amino)-3-oxo-2-(phenylselanyl)propanoate (1)

A THF (20 ml) solution of **8** (631 mg, 1.56 mmol) was added slowly at 0°C to a stirred suspension of NaH (68 mg, 60% mineral oil dispersion, 1.71 mmol) in THF (10 ml). After 20 min, phenylselenyl chloride (299 mg, 1.53 mmol) was added in one portion at −78°C. The reaction was stirred at −78°C for 2 h and then quenched with water. After removal of the solvent, the residue was extracted with Et_2O, washed with water, and dried over anhydrous Na_2SO_4. After it was filtered and concentrated, the crude product was purified by precooled column chromatography to give **1** (691 mg, 79%) as a yellow oil. Analytical TLC (silica gel 60), 50% EtOAc in *n*-hexane, $R_f = 0.56$; ^1H NMR (400 MHz, $CDCl_3$) δ 7.64–7.12 (m, 15H), 5.52 (dt, $J = 4.1$, 15.6 Hz, 0.85 × 1H), 5.44 (dt, $J = 4.6$, 15.6 Hz, 0.15 × 1H), 4.87 (dd, $J = 9.4$, 15.4 Hz, 1H), 4.63 (s, 0.27 × 1H), 4.61 (s, 0.77 × 1H), 3.90–3.67 (m, 2H), 3.69 (s, 3H), 2.28 (dt, $J = 6.0$, 8.7 Hz, 1H), 1.53 (dd, $J = 5.0$, 8.7 Hz, 1H), 1.48 (t_{obs}, $J = 5.5$ Hz, 1H), 1.22 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.7, 166.8, 146.3, 141.1, 135.9, 135.6, 133.0, 130.8, 129.1, 128.8, 128.7, 128.6, 128.4, 127.4, 126.8, 126.1, 58.3, 53.1, 50.4, 47.1, 37.3, 29.5, 28.3, 22.2; IR (neat) 2958, 1732, 1648 cm^{-1}; LRMS for $C_{32}H_{35}NO_3Se$ (EI, 20 eV) m/z 561 (M^+, 14); HRMS (EI) for $C_{32}H_{35}NO_3Se$ (M^+): calcd. 561.1782, found 561.1790.

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