2-Methylisoselenochromanium Salts: Spectroscopic Properties and Reactions

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Some 2-methylisoselenochromanium salts were prepared. Proton nuclear magnetic resonance spectra in $CDCl_3$ and electron impact mass spectra showed that the selenonium tosylate (4) and mesylate (5) are selenuranes and that the tetrafluoroborate (2) and triflate (3) are selenonium salts. Their structures at the selenium atom are influenced by their counter anions. Selenonium salts (2—5) reacted with some nucleophiles to give a styrene derivative (6) and methyl phenethyl selenides (7, 9).

Keywords selenurane; selenonium salt; isoselenochroman; β-elimination; ring-opening; demethylation

Stable tetravalent selenium compounds (selenuranes) such as selenonium tetrahalides, alkylselenenyl trihalides, dialkylselenide dihalides, tetraarylselenuranes, and tetraalkoxyselenuranes are well known.[1] However, trialkylselenuranes are generally unstable and easily converted into their ionic form (selenonium salts).[2] Selenurane has a trigonal bipyramidal center on selenium and the bonds consist of three sp^2- and two *p*-orbitals (form A in Fig. 1). On the other hand, the selenium center of a selenonium salt has 3 eq sp^3-bonds (form B in Fig. 1).

Previously, we reported on the reactivities of cyclic selenonium ylides stabilized by electron-withdrawing groups.[3-6] In this paper, we describe the synthesis of some isoselenochromanium salts having no functional groups and investigated their reactivities toward various nucleophiles.

Results and Discussion

2-Methylisoselenochromanium tetrafluoroborate (**2**) was prepared by the reactions of isoselenochroman (**1**)[6] with methyl iodide and silver tetrafluoroborate, or with dimethoxycarbenium tetrafluoroborate in 87% and quantitative yields, respectively. Other 2-methylisoselenochromanium sulfonates (**3**—**5**) were synthesized from **1** and the corresponding methyl sulfonates in quantitative, 74.5%, and 63% yields, respectively. The structures of **2**—**5** were established by their proton nuclear magnetic resonance (^{1}H-NMR) and mass spectra (MS). Their ^{1}H-NMR data are listed in Table I. The ^{1}H-NMR spectra of tetrafluoroborate **2** and trifluoromethanesulfonate (triflate) **3** in deuteriochloroform ($CDCl_3$) showed two singlets at δ *ca.* 4.4 assigned to Se-benzylic protons. Their electron impact MS (EI-MS) did not show the molecular ion peaks, but showed only fragment ion peaks arising from the cation moieties. These results indicate that compounds **2** and **3** take ionic forms (selenonium salts) and exist as isomeric mixtures due to pyramidal inversion at the selenium atom, with isomer ratios of about 1 : 1. On the other hand, the ^{1}H-NMR spectra of the *p*-toluenesulfonate (tosylate) **4** and the methanesulfonate (mesylate) **5** in $CDCl_3$ showed a pair of doublets at δ *ca.* 4.5 and 4.6 assigned to Se-benzylic protons with geminal coupling. The protons at the C(3)-position appeared as a pair of multiplets at δ *ca.* 2.7 and 4.2: only one proton was somewhat shifted downfield by the anisotropic effect of the sulfonyl group. Their EI-MS showed molecular ion peaks. Hence, they take selenurane structures having four covalent bonds. Furthermore, their ^{1}H-NMR spectra showed one singlet of methyl protons with chemical shifts lower than those of **2** and **3**. Consequently, methyl and sulfonyl groups were assigned to occupy the apical positions. In contrast, the ^{1}H-NMR spectra of **2**—**5** in a polar solvent, deuterioacetonitrile (CD_3CN), were almost the same as each other and similar to those of selenonium salts (**2**, **3**) in $CDCl_3$. These results indicate that selenuranes (**4**, **5**) are readily transformed to the ionic form in polar solvents, probably due to solvation.

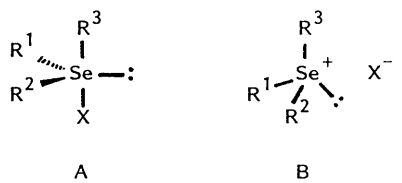

Fig. 1

Compounds **2**—**5** were thermally and photochemically very stable. Selenuranes **4** and **5** easily reacted with sodium tetrafluoroborate or triflic acid to give the corresponding selenonium salts (**2**, **3**). In contrast, the selenonium salts (**2**, **3**) could not be converted into selenuranes (**4**, **5**) on similar treatment. When the selenonium salt **2** was treated with triflic acid, an inseparable mixture of selenonium salts **2** and **3** was obtained. Treatment of **3** with sodium tetrafluoroborate similarly afforded a mixture of **2** and **3**. In the case of selenuranes, inseparable mixtures of selenuranes **4** and **5** were given by the reaction of **4** with methanesulfonic acid or the reaction of **5** with *p*-toluenesulfonic acid. We consider that the selenuranes are an unstable form of selenonium salts. The stable trialkylselenuranes are formed when the counter anions have weak nucleophilicity and small steric hindrance, because an increase of steric interaction between alkyl groups and their counter anions would be expected to favor the ionic form.

We next investigated the reactions of the compounds (**2**—**5**) with bases. Treatment of **2**—**5** with sodium hydride in several aprotic solvents gave 2-(methylselenomethyl)-styrene (**6**). The results are listed in Table II (entries 1—6). In the reaction of **3**, the yield of **6** was relatively high since the strong electron-withdrawing nature of the trifluoromethanesulfonyl group would make abstraction of the β-proton by base easy (entry 4). Differences of the other counter anions and solvents produced little difference in the reactions. The products other than **6** could not be isolated. This may be due to the instability of the ylide, which is formed by α-proton abstraction and may be converted to a complex mixture of products.

TABLE I. ^{1}H-NMR Spectra of 2-Methylisoselenochromanium Salts (**2**—**5**)

Compd.	Solvent	SeMe	1-H	3-H	4-H	ArH	Me
2	$CDCl_3$	2.20	4.37 4.38	2.67—2.80 3.15—3.17	3.31—3.33 4.06—4.13	7.33—7.44	
3	$CDCl_3$	2.21 2.17	4.42 4.43	2.60—2.71 3.10—3.22	3.27—3.34 4.11—4.18	7.26—7.42	
4	$CDCl_3$	2.26	4.53 (d, J=13 Hz) 4.59 (d, J=13 Hz)	2.61—2.72 4.16—4.23	3.15—3.19	7.15 (d, J=8 Hz) 7.73 (d, J=8 Hz) 7.21—7.36	2.34
5	$CDCl_3$	2.28	4.54 (d, J=13 Hz) 4.61 (d, J=13 Hz)	2.76—2.85 4.18—4.23	3.17—3.29	7.28—7.44	2.73
2	CD_3CN	2.13	4.23	2.74—2.83 2.96—3.07	3.29—3.38 3.72—3.80	7.37—7.42	
3	CD_3CN	2.13	4.24	2.73—2.77 2.82—3.08	3.29—3.38 3.73—3.80	7.37—7.48	
4	CD_3CN	2.11	4.33	2.67—2.77 3.01—3.12	3.24—3.32 3.79—3.87	7.17 (d, J=8 Hz) 7.65 (d, J=8 Hz) 7.37—7.39	2.43
5	CD_3CN	2.14	4.41	2.67—3.20	3.23—3.40 3.80—4.02	7.24—7.50	2.56

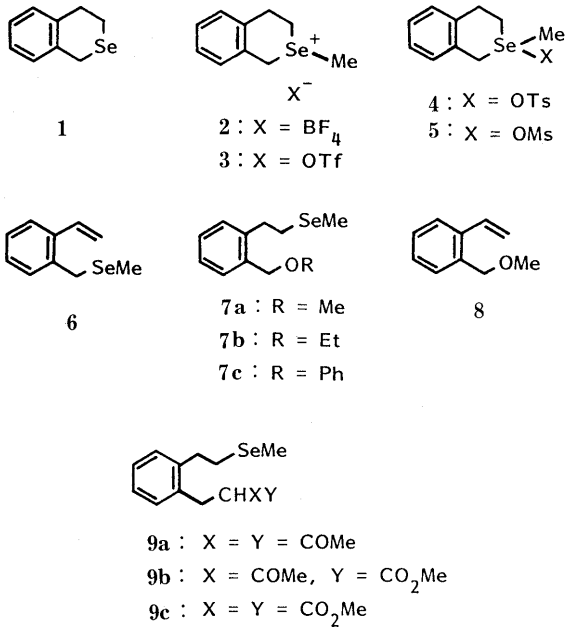

Fig. 2

TABLE II. Reactions of **2**—**5** with Bases

Entry	Starting material	Base	Solvent	Product (%)
1	**2**	NaH	CH_2Cl_2	**6** (10.1)
2	**2**	NaH	MeCN	**6** (29.4)
3	**2**	NaH	THF	**6** (29.1)
4	**3**	NaH	THF	**6** (45.5)
5	**4**	NaH	THF	**6** (14.5)
6	**5**	NaH	THF	**6** (14.5)
7	**2**	MeONa	MeOH	**6** (55.1), **7a** (22.5)
8	**2**	MeONa	C_6H_6	**6** (42.5), **7a** (7.5)
9	**2**	EtONa	EtOH	**6** (68.9), **7b** (14.1)
10	**2**	NaOH	H_2O	**6** (42.4)
11	**3**	MeONa	MeOH	**6** (51.7), **7a** (23.3)
12	**4**	MeONa	MeOH	**6** (58.2), **7a** (30.0)
13	**4**	MeONa	C_6H_6	**6** (61.8), **7a** (30.0)
14	**5**	MeONa	MeOH	**6** (47.1), **7a** (32.3)
15	**2**	PhONa	C_6H_6	**6** (5.9), **7c** (49.0)
16	**2**	PhONa	MeOH	**6** (31.1), **7a** (16.2), **7c** (33.9)
17	**3**	PhONa	C_6H_6	**6** (7.3), **7c** (64.3)
18	**4**	PhONa	C_6H_6	**6** (12.7), **7c** (64.3)
19	**5**	PhONa	C_6H_6	**6** (7.1), **7c** (55.6)

Next, we investigated the reactivities of **2**—**5** with sodium alkoxides or phenoxide (entries 7—19 in Table II). Reactions with sodium alkoxides afforded 2-(alkoxymethyl)-phenethyl methyl selenides (**7a, b**) and **6**. Since the styrene derivative (**6**) was mainly obtained, sodium alkoxides were supposed to serve as bases rather than the nucleophiles. For the determination of the structures of **7a, b**, **7a** was treated with hydrogen peroxide to give 2-(methoxymethyl)styrene (**8**) in 66% yield. The reaction of **2** with sodium hydroxide afforded only **6** without the benzyl alcohol derivative (entry 10). No difference of reactivities among **2**—**5** was observed. Reactions of **2**—**5** with reagents of stronger nucleophilic character such as sodium phenoxide afforded 2-(phenoxymethyl)phenethyl methyl selenide (**7c**) and **6** (entries 15—19). In all cases, the major product was the nucleophilic ring-opening product **7c**. The reaction of **2** with sodium phenoxide in methanol gave an intermediate result between entries 8 and 15; it produced **6** (31.1%), **7a** (16.2%), and **7c** (33.9%) (entry 16).

These results indicate that all the reactions proceed *via* the same transition state. Selenuranes (**4**, **5**) would be immediately converted into their ionic form and the alkoxide anions attack the benzylic position of the isoselenochromanium ion in a nucleophilic manner. Their counter anions are readily neutralized by sodium cation, and thus do not affect their reactivities. Ligand exchange on the selenium did not occur, either.[7]

Finally, the selenonium salt **2** reacted with carbanions which have moderate nucleophilicities. Reaction of **2** with sodium acetylacetonide in *N*,*N*-dimethylformamide (DMF) gave 2-(2,2-diacetylethyl)phenethyl methyl selenide (**9a**) and **1** in 42.1% and 10.1% yields, respectively. When tetrahydrofuran (THF) or ether was used as the solvent, the reactions proceeded very slowly. Product **9a** was a mixture of keto–enol tautomers with the isomer ratio of enol/keto = 1.25. Selenonium salt **2** reacted with sodium methyl acetoacetate or sodium dimethyl malonate to afford ring-opened prod-

ucts (**9b, c**) and **1**. Enolate anions mainly attacked the benzylic carbon of **2** without β-elimination. A part of **2** may react as a methylating reagent to give **1** but the methylated products were not obtained. Reaction of **2** with an enolate of acetophenone afforded only the styrene derivative **6** in 10.4% yield. Some Grignard reagents did not react with the selenonium salt but a new type of single electron transfer reduction proceeded.[8]

Experimental

Melting points were taken on a Yanagimoto micro melting point apparatus and are uncorrected. ^{1}H-NMR spectra were determined with a Hitachi R-20B (60 MHz) or a JEOL GX-270 (270 MHz) spectrometer and chemical shifts are given in parts per million relative to tetramethylsilane as an internal standard. MS were recorded with a JEOL JMS-D300 spectrometer and high-resolution MS with a JMA 2000 on-line system. Infrared (IR) spectra of solids (KBr) and liquids (film or CCl_4 solution) were measured with a JASCO IRA-100 spectrophotometer.

2-Methylisoselenochromanium Tetrafluoroborate (2) (a) Silver tetrafluoroborate (3.0 g, 17 mmol) was added to a solution of isoselenochroman (**1**)[6] (3.0 g, 15 mmol) in dichloromethane (30 ml) in the presence of methyl iodide (10.8 g, 75 mmol) at 0 °C and the mixture was stirred overnight at room temperature. The precipitate was filtered and washed with acetonitrile. The filtrate and washings were combined and the solvent was removed under reduced pressure. The residual solid was recrystallized from acetonitrile–ether to give colorless prisms (3.95 g, 88%), mp 125—126 °C. IR (KBr) cm^{-1}: 1030 (BF_4^-). MS *m/z*: 213 $(M-BF_4)^+$. *Anal.* Calcd for $C_{10}H_{13}BF_4Se$: C, 40.17; H, 4.38. Found: C, 40.00; H, 4.31. Its ^{1}H-NMR data are listed in Table I.

(b) A solution of boron trifluoride etherate (4.3 g, 30 mmol) in dry dichloromethane (6 ml) was carefully added to trimethyl orthoformate (3.2 g, 30 mmol) at −30 °C and then the mixture was stirred for 30 min. The temperature was gradually raised to 0 °C and then lowered again to −30 °C. A solution of **1** (3.0 g, 15 mmol) in dry dichloromethane (20 ml) was gradually added to the solution of dimethoxycarbenium tetrafluoroborate thus prepared and the mixture was stirred overnight at 0 °C. Ether was added to the reaction mixture. The precipitate that appeared was filtered off, washed with ether, and recrystallized from acetonitrile–ether to give **2** (4.5 g, 99%). This sample was identical with the specimen prepared by method (a).

2-Methylisoselenochromanium Triflate (3) Isoselenochroman (**1**) (500 mg, 2.5 mmol) was gradually added to methyl triflate (416 mg, 2.5 mmol) at 0 °C. A precipitate immediately appeared. It was filtered off, and recrystallized from acetonitrile–ether to give colorless prisms (908 mg, 99%), mp 122—123 °C. IR (KBr) cm^{-1}: 1245, 1265 (SO_3). MS *m/z*: 213 $(M-TfO)^+$. *Anal.* Calcd for $C_{11}H_{13}F_3O_3SSe$: C, 36.57; H, 3.63. Found: C, 36.58; H, 3.56. ^{1}H-NMR data are listed in Table I.

2-Methyl-2-*p*-tosyloxyisoselenochroman (4) A mixture of isoselenochroman (**1**) and an equimolar amount of methyl *p*-tosylate was heated at 60 °C and melted. The mixture was stirred at room temperature for 12—24 h until it solidified and the solid was recrystallized from dichloromethane–hexane gave colorless needles (74.5%), mp 125—126 °C. IR (KBr) cm^{-1}: 1195, 1210 (SO_3). MS *m/z*: 384 (M^+). *Anal.* Calcd for $C_{17}H_{20}O_3SSe \cdot 1/2H_2O$: C, 52.04; H, 5.39. Found: C, 51.80; H, 5.34. ^{1}H-NMR data were listed in Table I.

2-Mesyloxy-2-methylisoselenochroman (5) This compound was prepared in the same way as compound **4**. Recrystallization from dichloromethane–hexane gave colorless prisms (62.7%), mp 135—136 °C. IR (KBr) cm^{-1}: 1200, 1220 (SO_3). MS *m/z*: 308 (M^+). *Anal.* Calcd for $C_{11}H_{16}O_3SSe \cdot 1/2H_2O$: C, 42.38; H, 5.33. Found: C, 42.35; H, 5.17. ^{1}H-NMR data are listed in Table I.

Reactions of 4 and 5 with Sodium Tetrafluoroborate An equimolar amount of sodium tetrafluoroborate was added to a solution of selenurane (**4** or **5**) (100 mg) in methanol (5 ml) and water (5 drops) and the mixture was heated at 50 °C for 1 min. After the solution had cooled, it was dried over $MgSO_4$. The solution was filtered through Celite 545 and the filtrate was evaporated. The residual solid was recrystallized from acetonitrile–ether to give **2** (83.3% from **4**, 79.3% from **5**). This product was identical with an authentic specimen in terms of ^{1}H-NMR and IR spectra.

Reactions of 4, 5 with Trifluoromethanesulfonic Acid An equimolar amount of trifluoromethanesulfonic acid was added to a solution of a selenurane (**4** or **5**) (300 mg) in dry dichloromethane (15 ml) at 0 °C and the mixture was stirred for 20 min. The solvent was removed under reduced pressure and the residual solid was recrystallized from acetonitrile–ether to give **3** (88.4% from **4**, 80.0% from **5**). This product was identical with an authentic specimen in terms of ^{1}H-NMR and IR spectra.

Reactions of 2—5 with Bases A selenonium compound (**2—5**) (300 mg) was added to a suspension of an equimolar amount of base in an appropriate solvent (20 ml). The mixture was stirred for 1 h at room temperature under a nitrogen atmosphere and then refluxed for 7 h. The solvent was removed under reduced pressure and the residue was separated by preparative thin layer chromatography (TLC) on silica gel using hexane–dichloromethane (3 : 2). The products and their yields are summarized in Table II.

2-(Methylselenomethyl)styrene (**6**): A colorless oil. ^{1}H-NMR ($CDCl_3$) δ: 1.88 (3H, s, CH_3), 3.80 (2H, s, $ArCH_2$), 5.45 (1H, dd, J=11.0, 1.2 Hz, =CH_2), 5.65 (1H, dd, J=17.3, 1.2 Hz, =CH_2), 7.02 (1H, dd, J=17.3, 11.0 Hz, ArCH=), 7.10—7.70 (4H, m, ArH). High-resolution MS *m/z*: Calcd for $C_{10}H_{12}Se$: 212.0104. Found: 212.0105.

2-(Methoxymethyl)phenethyl Methyl Selenide (**7a**): A colorless oil. ^{1}H-NMR ($CDCl_3$) δ: 2.00 (3H, s, $SeCH_3$), 2.50—3.03 (4H, m, CH_2CH_2), 3.38 (3H, s, OCH_3), 4.48 (2H, s, OCH_2), 7.10—7.60 (4H, m, ArH). High-resolution MS *m/z*: Calcd for $C_{11}H_{16}OSe$: 244.0365. Found: 244.0357.

2-(Ethoxymethyl)phenethyl Methyl Selenide (**7b**): A colorless oil. ^{1}H-NMR ($CDCl_3$) δ: 1.23 (3H, t, J=6.8 Hz, CH_2CH_3), 2.00 (3H, s, $SeCH_3$), 2.50—3.25 (4H, m, CH_2CH_2), 3.55 (2H, q, J=6.8 Hz, CH_2CH_3), 4.54 (2H, s, $ArCH_2O$), 7.05—7.50 (4H, m, ArH). High-resolution MS *m/z*: Calcd for $C_{12}H_{18}OSe$: 258.0322. Found: 258.0547.

2-(Phenoxymethyl)phenethyl Methyl Selenide (**7c**): A colorless oil. ^{1}H-NMR ($CDCl_3$) δ: 1.93 (3H, s, CH_3), 2.60—3.40 (4H, m, CH_2CH_2), 5.09 (2H, s, $ArCH_2O$), 6.80—7.65 (9H, m, ArH). High-resolution MS *m/z*: Calcd for $C_{16}H_{18}OSe$: 306.0523. Found: 306.0544.

Reaction of 7a with Hydrogen Peroxide A 35% aqueous hydrogen peroxide solution (46 mg, 0.4 mmol) was gradually added to a solution of **7a** (100 mg, 0.4 mmol) in THF (10 ml) at 0 °C. The mixture was stirred for 4 h at 0 °C and for 4 h at room temperature, poured into water and extracted with dichloromethane. The dichloromethane layer was washed with water and dried over $MgSO_4$. The solvent was removed under reduced pressure and the residue was separated by preparative TLC on silica gel using hexane–dichloromethane (3 : 1) to give 2-(methoxymethyl)styrene (**8**) (40 mg, 66.0%) as a colorless oil. This sample was identical with an authentic specimen[9] in terms of ^{1}H-NMR, IR, and MS.

Reactions of 2 with Carbanions An active methylene compound (2.2 mmol) was added to a suspension of sodium hydride (48 mg, 2 mmol) in dry DMF (20 ml) and the mixture was stirred for 1 h at room temperature. The selenonium salt **2** (300 mg, 1 mmol) was added to the solution and the mixture was stirred for 2 d at room temperature under a nitrogen atmosphere, poured into water and extracted with benzene–hexane (4 : 1). The extract was washed with water and dried over $MgSO_4$. The solvent was removed under reduced pressure and the residue was separated by preparative TLC on silica gel using hexane–dichloromethane (2 : 1). Yields of products were as follows: **9a** (42.1%) and **1** (10.1%) from sodium acetylacetonide, **9b** (51.8%) and **1** (39.1%) from sodium methyl acetoacetate, and **9c** (44.2%) and **1** (10.1%) from sodium dimethyl malonate.

2-(2,2-Diacetylethyl)phenethyl Methyl Selenide (**9a**): A colorless oil. IR (CCl_4) cm^{-1}: 1700, 1730 (C=O), 1580—1620 (enol). ^{1}H-NMR ($CDCl_3$) δ (keto-form): 2.02 (6H, s, $CH_3 \times 2$), 2.13 (3H, s, $SeCH_3$), 2.73—2.83 (2H, m, CH_2Se), 2.99—3.19 (2H, m, $ArCH_2CH_2$), 3.63 (2H, d, J=7.3 Hz, $ArCH_2CH$), 4.03 (1H, t, J=7.3 Hz, CH), 7.01—7.27 (4H, m, ArH); δ (enol-form): 2.02 (3H, s, CH_3), 2.06 (3H, s, CH_3), 2.13 (3H, s, $SeCH_3$), 2.73—2.83 (2H, m, CH_2Se), 2.99—3.19 (2H, m, $ArCH_2CH_2$), 3.64 (2H, s, $ArCH_2C$=), 7.01—7.27 (4H, m, ArH), 16.86 (1H, s, OH). High-resolution MS *m/z*: Calcd for $C_{15}H_{20}O_2Se$: 312.0612. Found: 312.0627.

2-(2-Acetyl-2-methoxycarbonylethyl)phenethyl Methyl Selenide (**9b**): A colorless oil. IR (CCl_4) cm^{-1}: 1250, 1745 (ester), 1720 (C=O). ^{1}H-NMR ($CDCl_3$) δ: 2.02 (3H, s, $SeCH_3$), 2.19 (3H, s, $COCH_3$), 2.73—2.79 (2H, m, CH_2Se), 2.97—3.04 (2H, m, $ArCH_2CH_2$), 3.20 (2H, dd, J=7.3, 1.9 Hz, $ArCH_2CH$), 3.70 (3H, s, OCH_3), 3.81 (1H, t, J=7.3 Hz, CH), 7.09—7.26 (4H, m, ArH). High-resolution MS *m/z*: Calcd for $C_{15}H_{20}O_3Se$: 328.0576. Found: 328.0551.

2-(2,2-Dimethoxycarbonylethyl)phenethyl Methyl Selenide (**9c**): A colorless oil. IR (CCl_4) cm^{-1}: 1230, 1740 (ester). ^{1}H-NMR ($CDCl_3$) δ: 2.03 (3H, s, $SeCH_3$), 2.73—2.80 (2H, m, CH_2Se), 2.99—3.05 (2H, m, $ArCH_2CH_2$), 3.27 (2H, d, J=7.8 Hz, $ArCH_2CH$), 3.70 (6H, s, $OCH_3 \times 2$), 3.74 (1H, t, J=7.8 Hz, CH), 7.12—7.27 (4H, m, ArH). High-resolution MS *m/z*: Calcd for $C_{15}H_{20}O_4Se$: 344.0526. Found: 344.0503.

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