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Synthesis of 9-Oxoselenoxanthenium and Triarylselenonium Hexafluorophosphates

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Abstract—The selenoxides forming 9-oxoselenoxanthenium and triaryl selenonium hexafluorophosphates via interaction with heptyl phenyl ether and anisole in the $MeSO_3H–P_2O_5$ mixture followed by treatment with KPF_6 have been obtained via selenoxantene-9-one and diphenyl selenide derivatives oxidation with *m*-chloroperoxybenzoic acid or hydrogen peroxide.

Keywords: selenoxanthen-9-one, diaryl selenide, diaryl selenoxide, aryl(hetaryl)selenonium hexafluorophosphate

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Onium (iodonium, sulfonium, or ammonium) salts are used as initiators of cationic and radical photopolymerization [1–4]. The most common compounds of this class are triarylsulfonium salts [5–9] and sulfonium derivatives based on sulfur-containing heterocyclic compounds (thioxanthene, thianthrene, benzothiophene, etc.) [10–14]; they are interest in view of holography, acid generators for photoresists, and photochemical detritylation in microchip oligonucleotide synthesis applications.

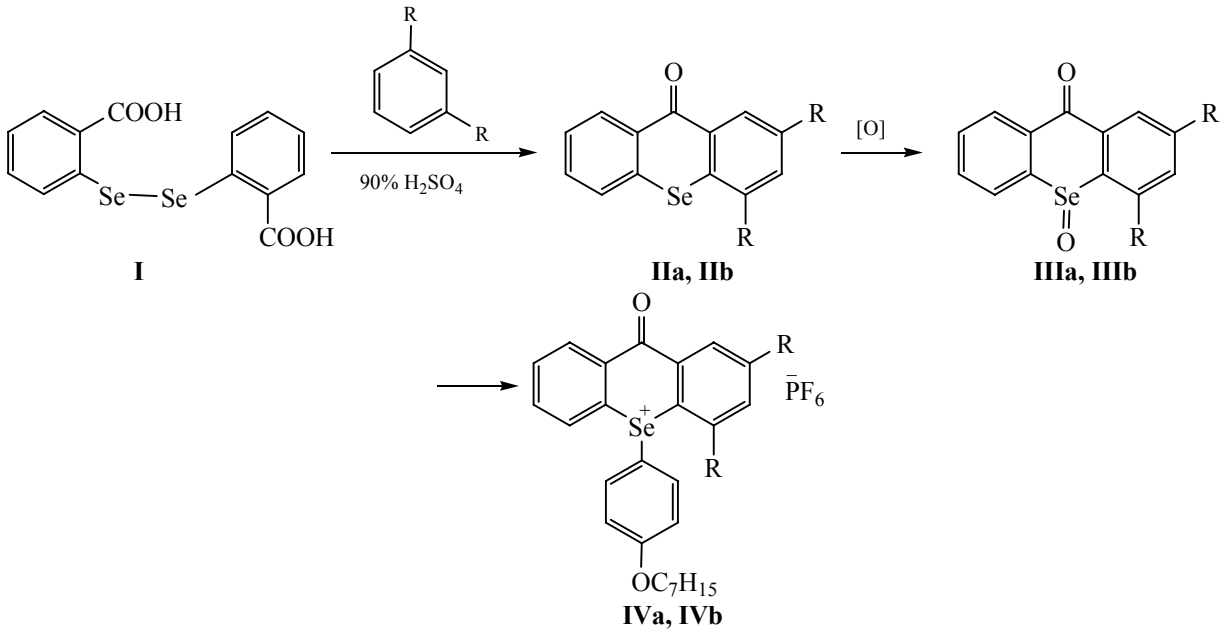
Introduction of heavy atoms in the photoinitiator molecule enhances the singlet-triplet $S_1–T_1$ conversion in the excited state [15, 16]. Since photochemical reactions often involve the triplet state of the reactant, substitution of sulfur in organic sulfonate salts with heavier atoms (for example, selenium or tellurium) can improve their photochemical activity.

We have earlier suggested application of thioxanthene hexafluorophosphate synthesized via reaction of S-oxides of substituted thioxanthene-9-ones with activated aromatic compounds as photoinitiators [17, 18]. This work aimed to extend the approach to prepare the selenonium salts with selenoxide derivatives of selenoxanthene-9-one and diaryl selenides as an example. To the best of our knowledge, the related information has been absent in the reference literature.

Similarly to dithiosalicylic acid [19], diselenosalicylic acid **I** reacted with aromatic hydrocarbons (benzene or *m*-xylene) in 90% sulfuric acid to formation of selenoxanthenones **IIa** and **IIb** that were further converted into the corresponding selenoxides (**IIIa** and **IIIb**) in the course of oxidation with *m*-chloroperoxybenzoic acid. Noteworthily, in contrast to the sulfur analogs, diaryl selenides are not overoxidized into selenones [20]; hence, formation of compounds **IIIa** and **IIIb** went smoothly and the yields were good. Reaction of the obtained selenoxides with heptyl phenyl ether in a 10 : 1 methanesulfonate–phosphoric anhydride mixture followed by treatment with aqueous solution of KPF_6 yielded selenoxanthenium hexafluorophosphates **IVa** and **IVb** (Scheme 1).

We further extended the above-described procedure to prepare triaryl selenonium derivatives. Those compounds have been better described in the literature as compared to the selenoxanthene salts. A classic method of their synthesis consists in the interaction of aromatic hydrocarbons with selenium dioxide or diarylseleniumdichloride in the presence of $AlCl_3$ (the Friedel–Crafts reaction) [21]. Triaryl selenonium compounds can be prepared in the absence of $AlCl_3$ as well, for example, upon heating of anisole and selenium dioxide in trifluoroacetic acid [22]. Other methods of triaryl selenonium salts synthesis are arylation of diaryl selenides with diaryl iodonium salts catalyzed by copper [23, 24] and condensation of organometallic compounds (Grignard reagent and others) with

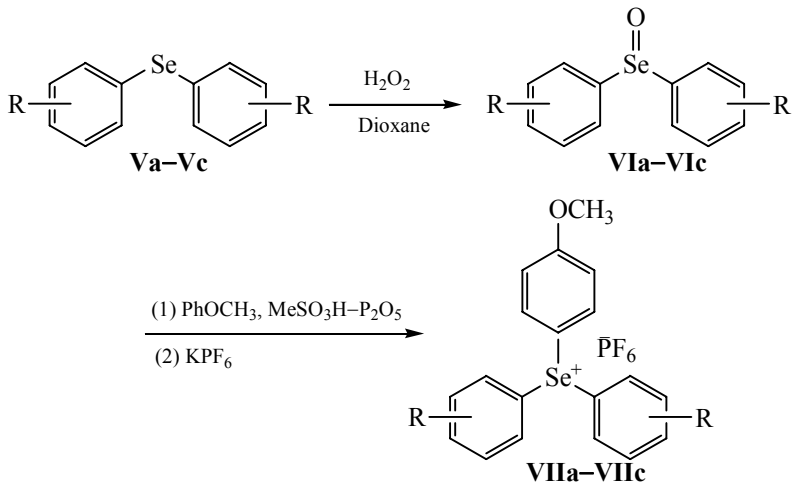
Scheme 1.



I **IIa, IIb** **IIIa, IIIb** **IVa, IVb**

R = H (**a**), CH_3 (**b**).

Scheme 2.



Va–Vc **VIa–VIc** **VIIa–VIIc**

R = H (**a**), 3-OCH_3 (**b**), 4-OCH_3 (**c**).

selenium oxochloride or diaryl selenoxides [25–28]. A significant drawback of those methods is the use of hardly accessible and insufficiently stable selenium derivatives [21]. Therefore, the method of synthesis aryl-substituted selenonium salts via the reaction of diaryl selenoxides with activated aromatic compounds seems advantageous.

Diphenyl selenide **Va** and its methoxy derivatives **Vb** and **Vc** converted into corresponding selenoxides **VIa–VIc** via oxidation with hydrogen peroxide were used as starting compounds. Their interaction with anisole in the $MeSO_3H–P_2O_5$ mixture followed by treatment with KPF_6 gave triarylselenonium hexafluorophosphates **VIIa–VIIc** in yields of 70–80% (Scheme 2).

Hexafluorophosphates **IV** and **VII** were colorless crystalline compounds soluble in organic solvents and insoluble in water.

Composition and structure of the products were confirmed by chemical analysis and spectral data. ^{1}H NMR spectrum of unsubstituted seleniumoxanthenone **IIa** contained weak-field signals of H^1 and H^8 protons at carbon atoms adjacent to the carbonyl group at 8.63 ppm. In the case of 2,4-dimethyl derivative **IIb**, the signal of the H^8 proton was found at 8.60 ppm, and the signal of H^1 proton revealed a slight upfield shift to 8.34 ppm, the signal position clearly showing deshielding influence of the carbonyl group; hence, the compound was identified as the 2,4-dimethyl derivative rather than the isomeric 1,3-disubstituted selenoxanthene.

In summary, reaction of selenoxide based on aryl (hetaryl) selenides with activated aromatic compounds (anisole or heptyl phenyl ether) in a $MeSO_3H$–P_2O_5 mixture is a simple and convenient method to prepare promising photoinitiators, aryl(hetaryl)selenonium salts.

EXPERIMENTAL

IR spectra of KBr pellets were recorded using a Vector 22 spectrometer. NMR spectra of $CDCl_3$ solutions were registered with a Bruker AV-300 [300.13 (^{1}H) and 282.36 MHz (F^{19})] or an AM-400 (400.13 MHz) spectrometers with residual solvent protons being the internal reference. ^{19}F chemical shifts are reported relative to the external C_6F_6 reference. Mass-spectra were registered using a Thermo Fisher Scientific DFS high-resolution mass spectrometer. The reaction progress and the products purity were evaluated by TLC on Sorbfil plates with chloroform as eluent. Silica gel (the 50–160 μm fraction) was used for column chromatography.

Bis(2-carboxyphenyl) diselenide (I) was obtained from anthranilic acid and potassium polyselenide [29]; mp 299–301°C (ethanol) (304–306°C [29], 296–297°C [30], and 358–360°C [31]). ^{1}H NMR spectrum coincided with the reference ones [29, 31]. Mass spectrum: *m/z* 397.8925 $[M]^+$ (calculated for $C_{14}H_{10}O_4{}^{78}Se_2$: *M* 397.8920).

9*H*-Selenoxanthene (IIa). 5 mL of anhydrous benzene was added to a mixture of 1 g of diselenide **I** and 25 mL of 90% H_2SO_4 cooled to 0°C, the mixture was stirred during 3 h, incubated at room temperature during 20 h, and then heated during 6 h at 50°C and during 25 h at 80–85°C. The mixture was cooled and poured onto ice. The precipitate was filtered off, washed sequentially with water, 5% aqueous NaOH, and water, and subject to chromatographic purification eluting with chloroform. Yield 0.5 g (38%), mp 190–192°C (191–192°C [32]). IR spectrum, ν, cm^{-1}: 1634 (C=O). ^{1}H NMR spectrum (400 MHz), δ, ppm: 7.45 and 7.52 (4H, $H^{2,3,6,7}$, $^3J_{HH}$ 8.0, $^4J_{HH}$ 2.0 Hz), 7.62 d.d (2H, $H^{4,5}$, $^3J_{HH}$ 8.0, $^4J_{HH}$ 2.0 Hz), 8.63 d.d (2H, $H^{1,8}$, $^3J_{HH}$ 8.0, $^4J_{HH}$ 2.0 Hz).

2,4-Dimethyl-9*H*-selenoxanthene-9-one (IIb) was prepared similarly from 1 g of diselenide **I** and 3 mL of *m*-xylene; heating at 130–135°C during 25 h. Yield 0.5 g (35%), mp 128–131°C. IR spectrum, ν, cm^{-1}: 3061, 2920, 2853, 2737 (C–H), 1630 (C=O), 1587 (C=C). ^{1}H NMR spectrum (400 MHz), δ, ppm: 2.42 s and 2.46 s (6H, 2Me), 7.28 d (1H, H^3, $^4J_{HH}$ 2.0 Hz), 7.44 t.d and 7.51 t.d (2H, $H^{6,7}$, $^3J_{HH}$ 8.0, $^4J_{HH}$ 1.5 Hz), 7.64 d.d (1H, H^5, $^3J_{HH}$ 8.0, $^4J_{HH}$ 1.5 Hz), 8.34 d (1H, H^1, $^4J_{HH}$ 2.0 Hz), 8.60 d.d (1H, H^8, $^3J_{HH}$ 8.0, $^4J_{HH}$ 1.5 Hz). Mass spectrum: *m/z* 284.0078 $[M]^+$ (calculated for $C_{15}H_{12}O^{76}Se$: *M* 284.0075).

Oxidation of selenoxanthenes (II) with *m*-chloroperbenzoic acid. A solution of 4 mmol *m*-chloroperbenzoic acid in 15 mL of chloroform was added to a cooled (0°C) solution of 2 mmol of selenoxanthene **II** in 10 mL of chloroform. The mixture was stirred during 30 min, washed with 5% aqueous NaOH and with water. The solution in chloroform was dried, about 20 mL of the solvent was distilled off, diethyl ether was added to the residue, and precipitate of the oxide **III** was separated off.

9*H*-Selenoxanthene-9-one-10-oxide (IIIa). Yield 64%, mp 237 to 240°C (mp 236–237°C [32]). IR spectrum, ν, cm^{-1}: 1666 (C=O), 806 (Se=O) (805 [33]). ^{1}H NMR spectrum (400 MHz), δ, ppm: 7.72 t.d and 7.80 t.d (4H, $H^{2,3,6,7}$, $^3J_{HH}$ 8.0, $^4J_{HH}$ 1.3 Hz), 8.08 d.d (2H, $H^{4,5}$, $^3J_{HH}$ 8.0, $^4J_{HH}$ 1.3 Hz), 8.38 d.d (2H, $H^{1,8}$, $^3J_{HH}$ 8.0, $^4J_{HH}$ 1.3 Hz). Mass spectrum: *m/z* 273.9687 $[M]^+$ (calculated for $C_{13}H_8O_2{}^{78}Se$: *M* 273.9692).

2,4-Dimethyl-9*H*-selenoxanthene-9-one-10-oxide (IIIb). Yield 87%, mp 198–200°C. IR spectrum, ν, cm^{-1}: 3055–2730 (C–H), 1657 (C=O), 800 (Se=O). ^{1}H NMR spectrum (400 MHz), δ, ppm: 2.45 s and 2.82 s (6H, 2Me), 7.39 d (1H, H^3, $^4J_{HH}$ 2.0 Hz), 7.71 t.d and 7.78 t.d (2H, $H^{6,7}$, $^3J_{HH}$ 8.0, $^4J_{HH}$ 1.5 Hz), 8.00 d.d (^{1}H, H^5, $^3J_{HH}$ 8.0, $^4J_{HH}$ 1.5 Hz), 8.13 d (1H, H^1, $^4J_{HH}$ 2.0 Hz), 8.41 d.d (1H, H^8, $^3J_{HH}$ 8.0, $^4J_{HH}$ 1.5 Hz). The mass spectrum, *m/z* (I_{rel}, %): 302 (1.74), 300 (0.77), 286 (35.51), 284 (13.01), 208 (100.00). Found, %: C 59.13; H 3.76. $C_{15}H_{12}O^{78}Se$. Calculated, %: C 59.40; H 3.96.

Diphenyl selenide (Va) was prepared from aniline and potassium polyselenide [34], bp 165–167°C (12 mmHg) {bp 165–167°C (12 mmHg) [34]}.

Bis(3-methoxyphenyl)selenide (Vb) was prepared similarly from *m*-anisidine and potassium polyselenide. Yield 30%, bp 169–171°C (1 mmHg) {bp 169°C (1 mmHg) [35]}.

Bis(4-methoxyphenyl)selenide (Vc) was obtained from anisole and $SeCl_2$ [36]. Yield 90%, mp 54–55°C (mp 54–55°C [20]).

Diaryl selenoxides (VIa–VIc) were prepared via oxidation of diaryl selenides **Va–Vc** with H_2O_2 (d = 1.145 g/mL) as described in [37].

Diphenyl selenoxide (VIa). Yield 86%, mp 110°C (mp 109°C [38]).

Bis(3-methoxyphenyl) selenoxide (VIb). Yield 61%, mp 131–132°C (precipitated with diethyl ether from methylene chloride). IR spectrum, ν, cm^{-1}: 829 (Se=O). ^1H NMR spectrum (300 MHz), δ, ppm.: 3.79 s (6H, 2OMe), 6.94 d.d. d (2H, $H^{4,4'}$, $^3J_{HH}$ 7.8, $^4J_{HH}$ 2.7, 1.2 Hz), 7.18 d. t (2H, $H^{6,6'}$, $^3J_{HH}$ 7.8, $^4J_{HH}$ 1.2 Hz), 7.27 d.d (2H, $H^{2,2'}$, ^4J 2.7, 1.2 Hz), 7.32 t (2H, $H^{5,5'}$, $^3J_{HH}$ 7.8 Hz). Found, %: C 54.15; H 4.56; Se 25.74. $C_{14}H_{14}O_3Se$. Calculated, %: C 54.39; H 4.56; Se 25.53.

Bis(4-methoxyphenyl) selenoxide (VIc). Yield 67%, mp 144°C (mp 144°C [37]).

Interaction of selenoxides of selenoxanthenone (III) with heptyl phenyl ether. A mixture of 1.5 mmol of selenoxide **III**, 1.8 mmol of heptyl phenyl ether, and 5 mL of a P_2O_5–$MeSO_3H$ (1 : 10) mixture was stirred at 20°C during 5 h, incubated during 20 h without stirring, cooled with icy water, and a solution of 2 mmol of KPF_6 in 5 mL of water was added dropwise. The mixture was stirred during 30 min and extracted with CH_2Cl_2. The organic layer was washed with aqueous solution of KPF_6 and water, evaporated, and treated with diethyl ether; precipitate of hexafluorophosphate **IV** was separated.

9-Oxo-10-(4-heptyloxyphenyl)-9*H*-selenoxantenium hexafluorophosphate (IVa). Yield 75%, mp 66–68°C. IR spectrum, ν, cm^{-1}: 2930, 2856 (C–H), 1668 (C=O), 841, 557 (P–F). ^1H NMR spectrum, δ, ppm: 0.86 m (3H, Me), 1.25 m (8H, $4CH_2$), 1.71 m (2H, CH_2), 3.92 m (2H, OCH_2), 6.93 d and 7.49 d (4H, C_6H_4, $^3J_{HH}$ 8.8 Hz), 7.89 m (4H, $H^{2,3,6,7}$), 8.00 m (2H, $H^{4,5}$), 8.61 m (2H, $H^{1,8}$). ^{19}F NMR spectrum, δ_F, ppm: 93.9 d (J_{FP} 672 Hz). Found, %: C 52.49; H 4.50; F 18.79. $C_{26}H_{27}O_2Se{\cdot}PF_6$. Calculated, %: C 52.44; H 4.54; F 19.16.

2,4-Dimethyl-9-oxo-10-(4-hepthyloxyphenyl)-9*H*-selenoxantenium hexafluorophosphate (IVb). Yield 84%, mp 149–153°C. IR spectrum, ν, cm^{-1}: 2953, 2928, 2856 (C–H), 1663 (C=O), 841, 557 (P–F). ^1H NMR spectrum, δ, ppm: 0.86 m (3H, $\underline{CH_3}CH_2$), 1.25 m (8H, $4CH_2$), 1.69 m (2H, CH_2), 2.53 s and 2.61 s (6H, 2Me), 3.89 m (2H, OCH_2), 6.90 d and 7.45 d (4H, C_6H_4, $^3J_{HH}$ 9.0 Hz), 7.55 d (1H, H^3, $^4J_{HH}$ 2.0 Hz), 7.85 m (2H, $H^{6,7}$), 8.17 m (1H, H^5), 8.34 (1H, H^1, $^4J_{HH}$ 2.0 Hz), 8.56 m (1H, H^8). ^{19}F NMR spectrum, δ_F, ppm: 90.5 d (J_{FP} 711 Hz). Found, %: F 18.40; Se 12.88. $C_{28}H_{31}O_2Se{\cdot}PF_6$. Calculated, %: F 18.30; Se 12.68.

Diphenyl(4-methoxyphenyl)selenonium, bis(3-methoxyphenyl)(4-methoxyphenyl)selenonium, and tris-(4-methoxyphenyl)selenonium hexafluorophosphates (VIIa–VIIc) were prepared similarly from 1 mmol of selenoxide **VI** and 1.5 mmol of anisole.

Compound VIIa. Yield 77%, mp 131–132°C (precipitated with diethyl ether from methylene chloride). IR spectrum, ν, cm^{-1}: 839, 557 (P–F). ^1H NMR spectrum (300 MHz), δ, ppm: 3.85 s (3H, OMe), 7.12 d (2H, $H^{3,5}$, $^3J_{HH}$ 9.0 Hz), 7.49 m (6H, H^{Ar}), 7.64 m (6H, H^{Ar}). ^{19}F NMR spectrum, δ_F, ppm: 89.9 d (J_{FP} 758 Hz). Found, %: F 23.48; Se 16.4. $C_{19}H_{17}OSe{\cdot}PF_6$. Calculated, %: F 23.49; Se 16.3.

Compound VIIb. Yield 71%, mp 152–153°C (precipitated with diethyl ether from methylene chloride). IR spectrum, ν, cm^{-1}: 843, 557 (P–F). ^1H NMR spectrum (300 MHz), δ, ppm: 3.62 s (6H, 2OMe), 3.69 s (3H, OMe), 6.83 d.d. d (2H, $H^{4',4''}$, $^3J_{HH}$ 8.2, $^4J_{HH}$ 2.0, 0.2 Hz), 6.94 t (2H, $H^{2',2''}$, $^4J_{HH}$ 2.0 Hz), 6.96 d (2H, $H^{3,5}$, $^3J_{HH}$ 9.0 Hz), 7.02 d.d. d (2H, $H^{6',6''}$, $^3J_{HH}$ 8.2, $^4J_{HH}$ 2.0, 0.8 Hz), 7.33 d (2H, $H^{2,6}$, $^3J_{HH}$ 9.0 Hz), 7.35 t (2H, $H^{5',5''}$, $^3J_{HH}$ 8.2 Hz). ^{19}F NMR spectrum, δ_F, ppm: 89.3 d (J_{FP} 757 Hz). Found, %: C, 46.69; H 3.92; F 21.02; Se 14.47. $C_{21}H_{21}O_3Se{\cdot}PF_6$. Calculated, %: C 46.24; H 3.85; F 20.90; Se 14.49.

Compound VIIc. Yield 53%, mp 60°C (ethanol). IR spectrum, ν, cm^{-1}: 843, 557 (P–F). ^1H NMR spectrum (300 MHz), δ, ppm: 3.86 s (9H, 3OMe), 7.09 d (6H, H^{Ar}, $^3J_{HH}$ 9.0 Hz), 7.42 d (6H, H^{Ar}, $^3J_{HH}$ 9.0 Hz). ^{19}F NMR spectrum, δ_F, ppm: 89.7 d (J_{FP} 771 Hz). Found, %: C, 45.59; H 3.92; F 21.09; Se 13.91. $C_{21}H_{21}O_3Se{\cdot}PF_6$. Calculated, %: C, 46.24; H 3.85; F 20.90; Se 14.52.

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