

SHORT  
COMMUNICATIONS

## Synthesis of 2,6-Dibromo-9-selenabicyclo[3.3.1]nonane-Based Pyridinium Salts Containing Acetal Groups

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**Abstract**—An efficient one-pot procedure has been developed for the synthesis of previously unknown 1,1'-(9-selenabicyclo[3.3.1]nonane-2,6-diyl)dipyridinium dibromides containing acetal moieties via transannular addition of selenium dibromide to *Z,Z*-cycloocta-1,5-diene, followed by reaction with pyridine-3- and 4-carbaldehyde acetals. The yields of the target products range from 80 to 97%.

**Keywords:** selenium dibromide, cycloocta-1,5-diene, pyridinecarbaldehyde acetals, pyridinium salts, selenabicyclo[3.3.1]nonane

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The chemistry and biological activity of organo-selenium compounds attract interest from scientists all over the world. A number of monographs and reviews have recently been published on the chemistry of organoselenium compounds and their biological activity, which demonstrate their high potential for the development of new medicines [1–6]. For instance, the selenium-containing drug ebselen has passed clinical trials as an anti-inflammatory drug, as well as for the treatment and prevention of cardiovascular diseases and ischemic stroke [7–9]. Ebselen has recently been found to exhibit a high anti-COVID-19 activity [8, 9]. Taking into account the need to vaccinate the population against coronavirus, which is an important way to protect against COVID-19, the development of new drugs for metabolic correction is an important problem. Metabolic correction can significantly reduce side effects caused by vaccination.

It was found that water soluble pyridinium salt **1** derived from 2,6-dibromo-9-selenabicyclo[3.3.1]nonane is promising for metabolic correction after vaccination [10, 11]. Administration of compound **1** in experimental animals significantly inhibits the development of pathological responses after injection of a tularemia vaccine and reduces the reactogenicity of brucellosis vaccine [10]. Moreover, compound **1** is nontoxic [10], and it does not exhibit glutathione peroxidase-like activity, i.e., it acts as a catalyst for

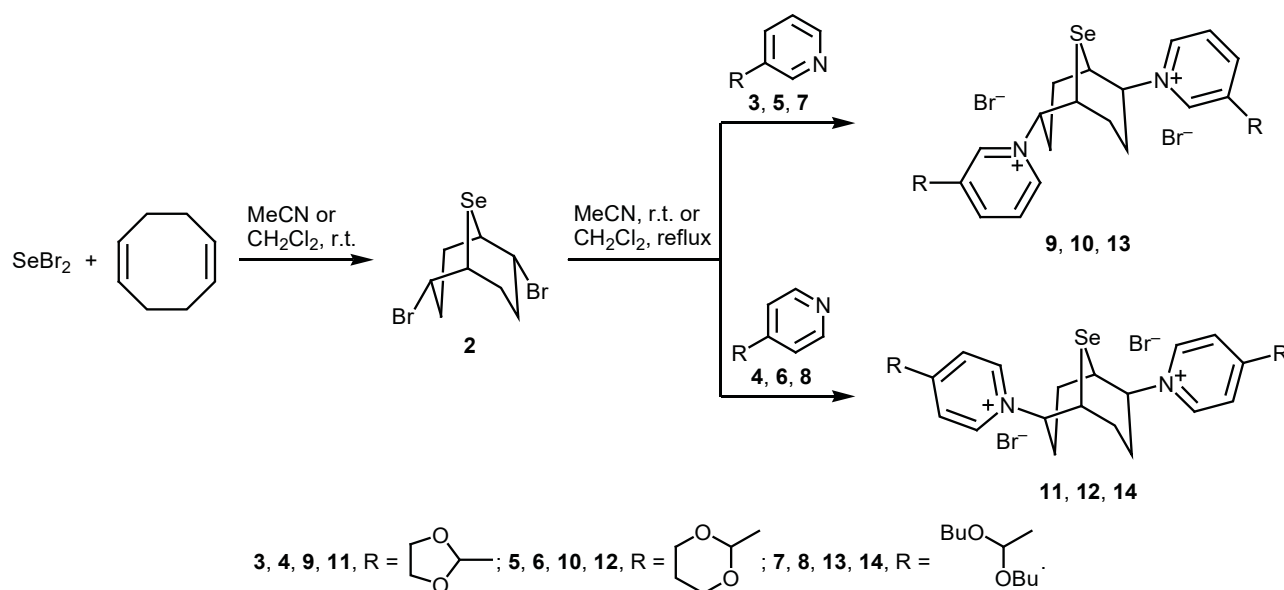
the decomposition of peroxide compounds in the organism [11].

Oxidative stress is one of the possible undesirable postvaccination responses of an organism, which develops as a result of increased generation of active oxygen species in cells. This may also lead to inflammatory and allergic reactions based on lipid peroxidation.

We previously described transannular addition of selenium dihalides to *Z,Z*-cycloocta-1,5-diene, which produced 2,6-dihalo-9-selenabicyclo[3.3.1]nonanes in nearly quantitative yield [12, 13]. Several derivatives of **1** were synthesized by reacting 2,6-dibromo-9-selenabicyclo[3.3.1]nonane (**2**) with substituted pyridines in boiling acetonitrile [14].

The present work was aimed at further developing this line of research and elaborating an efficient protocol for the synthesis of previously unknown series of water-soluble pyridinium salts containing acetal groups from 2,6-dibromo-9-selenabicyclo[3.3.1]nonane; these salts are interesting as potential agents for metabolic correction after vaccination. It is known that pyridinium salts containing various functional groups exhibit diverse biological activities such as anticancer, antiviral, antibacterial, and antifungal [15–17]. Furthermore, some pyridinium salts having acetal groups showed a high antiparasitic activity [16, 17].

Scheme 1.



As starting materials, we selected the following pyridines containing an acetal moiety: 3- and 4-(1,3-dioxolan-2-yl)pyridines **3** and **4**, 3- and 4-(1,3-dioxan-2-yl)pyridines **5** and **6**, and 3- and 4-(dibutoxymethyl)pyridines **7** and **8**. Acetals **3–8** were synthesized in 81–90% yield by reactions of pyridine-3- and -4-carbaldehydes with ethylene glycol, propane-1,3-diol, and butan-1-ol according to the reported procedure [18].

When dibromide **2** and substituted pyridine **3–8** were heated in boiling acetonitrile for a few hours, the target compound was formed, but the reaction was accompanied by side processes, in particular deacetalization whose contribution was most appreciable in the reactions with (dibutoxymethyl)pyridines **7** and **8**.

We found that dibromide **2** efficiently reacted with pyridines **3–6** in acetonitrile at room temperature when the reaction time was prolonged to 40 h. In this case, the yields of target products **9–12** reached 91–97% (Scheme 1).

We also developed a one-pot procedure for the synthesis of compounds **9–12** in 85–92% yields from selenium dibromide, *Z,Z*-cycloocta-1,5-diene, and pyridines **3–6**. After completion of the transannular addition of  $\text{SeBr}_2$  to *Z,Z*-cycloocta-1,5-diene in acetonitrile, pyridine **3–6** was added to the reaction mixture, and the mixture was stirred at room temperature for 45 h. Compounds **9–12** precipitated from the mixture and were filtered off and dried under reduced pressure (Scheme 1).

In the reaction with dibutyl acetals **7** and **8** under similar conditions, the corresponding pyridinium salts

did not precipitate from the reaction mixtures. According to the NMR data, the residue obtained after removal of the solvent contained the target pyridinium salt contaminated with by-products. We succeeded in selectively obtaining compounds **13** and **14** by carrying out the reaction in methylene chloride. Following a one-pot procedure, dibromide **2** was synthesized from selenium dibromide and *Z,Z*-cycloocta-1,5-diene in methylene chloride at room temperature, pyridine **7** or **8** was then added, and the mixture was heated under reflux ( $\sim 40^\circ\text{C}$ ) for 8 h. The solvent was distilled off, and the residue was washed with hexane and dried under reduced pressure. We thus isolated compounds **13** and **14** in 80–82% yield (Scheme 1).

The highest yields of the target products were obtained in the reactions with (1,3-dioxolan-2-yl)pyridines **3** and **4**, and the lowest yields (80–82%), from dibutyl acetals **7** and **8**. Furthermore, the yields of 3-substituted pyridine derivatives **9**, **10**, and **13** were slightly higher than the yields of the corresponding 4-substituted analogs **11**, **12**, and **14**.

**Reaction of dibromide 2 with pyridines 3–6 (general procedure).** Dibromide **2** (0.174 g, 0.5 mmol) was added to a solution of pyridine **3–6** (1 mmol) in acetonitrile (3 mL), and the mixture was stirred at room temperature for 40 h. The precipitate was filtered off through a Schott glass filter and dried under reduced pressure to a constant weight. The yields of **9**, **10**, **11**, and **12** were 97, 94, 93, and 91%, respectively.

**One-pot synthesis of compounds 9–12 (general procedure).** A solution of bromine (0.16 g, 1 mmol) in

acetonitrile (1 mL) was added to a mixture of elemental selenium (0.079 g, 1 mmol) and acetonitrile (1 mL), and the mixture was stirred until it became homogeneous. The resulting solution of selenium dibromide (1 mmol) in acetonitrile (2 mL) was added dropwise with stirring over a period of 5 min to a solution of *Z,Z*-cycloocta-1,5-diene (0.108 g, 1 mmol) in acetonitrile (6 mL). The mixture was stirred at room temperature for 2 h, a solution of pyridine **3–6** (2 mmol) in acetonitrile (1 mL) was added, and the mixture was stirred at room temperature for 45 h. The precipitate was filtered off through a Schott glass filter and dried under reduced pressure until constant weight. The yields of **9**, **10**, **11**, and **12** were 92, 90, 87, and 85%, respectively.

**One-pot synthesis of compounds 13 and 14 (general procedure).** A solution of bromine (0.16 g, 1 mmol) in methylene chloride (1 mL) was added to elemental selenium (0.079 g, 1 mmol), and the mixture was stirred until it became homogeneous. The resulting solution of selenium dibromide (1 mmol) in methylene chloride (1 mL) was added dropwise with stirring over a period of 10 min to a solution of *Z,Z*-cycloocta-1,5-diene (0.108 g, 1 mmol) in methylene chloride (5 mL). The mixture was stirred at room temperature for 3 h, a solution of pyridine **7** or **8** (2 mmol) in methylene chloride (1 mL) was added, and the mixture was refluxed with stirring for 8 h. The solvent was distilled off on a rotary evaporator, and the residue was washed with cold hexane on a Schott glass filter and dried under reduced pressure until constant weight. The yields of **13** and **14** were 82 and 80%, respectively.

**1,1'-(9-Selenabicyclo[3.3.1]nonane-2,6-diyl)bis[3-(1,3-dioxolan-2-yl)pyridinium] dibromide (9).** White powder, mp 105–106°C. <sup>1</sup>H NMR spectrum,  $\delta$ , ppm: 2.45–2.53 m (2H, CH<sub>2</sub>), 2.61–2.71 m (4H, CH<sub>2</sub>), 3.23–3.35 m (2H, CH<sub>2</sub>), 3.51–3.58 m (2H, CHSe), 4.16–4.25 m (8H, OCH<sub>2</sub>), 5.97–6.06 m (2H, CH<sub>2</sub>CHN), 6.24 s (2H, CHO), 8.26–8.31 m (2H, H<sub>py</sub>), 8.75–8.80 m (2H, H<sub>py</sub>), 9.19–9.25 m (4H, H<sub>py</sub>). <sup>13</sup>C NMR spectrum,  $\delta_C$ , ppm: 25.1 (CH<sub>2</sub>), 27.8 (CH<sub>2</sub>), 29.1 (CHSe), 65.3 (CH<sub>2</sub>O), 74.7 (CH<sub>2</sub>CHN), 98.9 (HCO), 128.6 (CH<sub>py</sub>), 139.5 (C<sub>py</sub>), 141.6 (CH<sub>py</sub>), 143.2 (CH<sub>py</sub>), 144.3 (CH<sub>py</sub>). Found, %: C 44.28; H 4.81; Br 24.78; N 4.24; Se 12.39. C<sub>24</sub>H<sub>30</sub>Br<sub>2</sub>N<sub>2</sub>O<sub>4</sub>Se. Calculated, %: C 44.40; H 4.66; Br 24.61; N 4.31; Se 12.16.

**1,1'-(9-Selenabicyclo[3.3.1]nonane-2,6-diyl)bis[3-(1,3-dioxan-2-yl)pyridinium] dibromide (10).** White powder, mp 124–125°C. <sup>1</sup>H NMR spectrum,  $\delta$ , ppm: 1.61–1.67 m (2H, CH<sub>2</sub>CH<sub>2</sub>O), 2.16–2.27 m (2H, CH<sub>2</sub>CH<sub>2</sub>O), 2.43–2.51 m (2H, CH<sub>2</sub>), 2.60–2.69 m (4H,

CH<sub>2</sub>), 3.22–3.34 m (2H, CH<sub>2</sub>), 3.52–3.56 m (2H, CHSe), 4.16–4.24 m (4H, OCH<sub>2</sub>), 4.33–4.39 m (4H, OCH<sub>2</sub>), 5.95–6.04 m (2H, CHN), 6.02 s (2H, CHO), 8.25–8.30 m (2H, H<sub>py</sub>), 8.72–8.77 m (2H, H<sub>py</sub>), 9.18–9.21 m (4H, H<sub>py</sub>). <sup>13</sup>C NMR spectrum,  $\delta_C$ , ppm: 25.9 (CH<sub>2</sub>CH<sub>2</sub>O), 26.3 (CH<sub>2</sub>), 29.1 (CH<sub>2</sub>), 30.4 (CHSe), 68.8 (CH<sub>2</sub>O), 76.1 (CHN), 98.0 (HCO), 129.8 (CH<sub>py</sub>), 142.2 (C<sub>py</sub>), 142.5 (CH<sub>py</sub>), 144.5 (CH<sub>py</sub>), 145.1 (CH<sub>py</sub>). Found, %: C 46.35; H 5.19; Br 23.84; N 4.27; Se 11.88. C<sub>26</sub>H<sub>34</sub>Br<sub>2</sub>N<sub>2</sub>O<sub>4</sub>Se. Calculated, %: C 46.10; H 5.06; Br 23.59; N 4.14; Se 11.66.

**1,1'-(9-Selenabicyclo[3.3.1]nonane-2,6-diyl)bis[4-(1,3-dioxolan-2-yl)pyridinium] dibromide (11).** White powder, mp 135–136°C. <sup>1</sup>H NMR spectrum,  $\delta$ , ppm: 2.29–2.41 m (2H, CH<sub>2</sub>), 2.48–2.61 m (4H, CH<sub>2</sub>), 3.23–3.30 m (2H, CH<sub>2</sub>), 3.46–3.51 m (2H, CHSe), 4.19–4.29 m (8H, OCH<sub>2</sub>), 6.01 s (2H, CHO), 5.98–6.03 m (2H, CH<sub>2</sub>CHN), 8.27 d (4H, H<sub>py</sub>, *J* = 6.3 Hz), 8.95 d (4H, H<sub>py</sub>, *J* = 6.3 Hz). <sup>13</sup>C NMR spectrum,  $\delta_C$ , ppm: 24.2 (CH<sub>2</sub>), 26.9 (CH<sub>2</sub>), 29.8 (CHSe), 63.4 (OCH<sub>2</sub>), 74.4 (CHN), 99.8 (HCO), 125.7 (CH<sub>py</sub>), 141.6 (CH<sub>py</sub>), 157.6 (C<sub>py</sub>). Found, %: C 44.22; H 4.72; Br 24.95; N 4.28; Se 12.36. C<sub>24</sub>H<sub>30</sub>Br<sub>2</sub>N<sub>2</sub>O<sub>4</sub>Se. Calculated, %: C 44.40; H 4.66; Br 24.61; N 4.31; Se 12.16.

**1,1'-(9-Selenabicyclo[3.3.1]nonane-2,6-diyl)bis[4-(1,3-dioxan-2-yl)pyridinium] dibromide (12).** White powder, mp 85–86°C. <sup>1</sup>H NMR spectrum,  $\delta$ , ppm: 1.61–1.67 m (2H, CH<sub>2</sub>CH<sub>2</sub>O), 2.13–2.23 m (2H, CH<sub>2</sub>CH<sub>2</sub>O), 2.43–2.51 m (2H, CH<sub>2</sub>), 2.61–2.69 m (4H, CH<sub>2</sub>), 3.14–3.27 m (2H, CH<sub>2</sub>), 3.50–3.56 m (2H, CHSe), 4.16–4.25 m (4H, OCH<sub>2</sub>), 4.31–4.39 m (4H, OCH<sub>2</sub>), 5.97 s (2H, CHO), 5.95–6.01 m (2H, CH<sub>2</sub>CHN), 8.25 d (4H, H<sub>py</sub>, *J* = 6.1 Hz), 9.17 d (4H, H<sub>py</sub>, *J* = 6.1 Hz). <sup>13</sup>C NMR spectrum,  $\delta_C$ , ppm: 24.7 (CH<sub>2</sub>CH<sub>2</sub>O), 25.2 (CH<sub>2</sub>), 27.8 (CH<sub>2</sub>), 29.0 (CHSe), 67.6 (OCH<sub>2</sub>), 74.3 (CHN), 97.1 (HCO), 125.5 (CH<sub>py</sub>), 143.4 (CH<sub>py</sub>), 155.5 (C<sub>py</sub>). Found, %: C 45.94; H 4.91; Br 23.36; N 3.98; Se 11.91. C<sub>26</sub>H<sub>34</sub>Br<sub>2</sub>N<sub>2</sub>O<sub>4</sub>Se. Calculated, %: C 46.10; H 5.06; Br 23.59; N 4.14; Se 11.66.

**1,1'-(9-Selenabicyclo[3.3.1]nonane-2,6-diyl)bis[3-(dibutoxymethyl)pyridinium] dibromide (13).** White powder, mp 55–56°C. <sup>1</sup>H NMR spectrum,  $\delta$ , ppm: 0.88–0.91 m (12H, CH<sub>3</sub>), 1.36–1.43 m (8H, CH<sub>2</sub>CH<sub>3</sub>), 1.62–1.68 m (8H, CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>), 2.47–2.56 m (2H, CH<sub>2</sub>), 2.60–2.73 m (4H, CH<sub>2</sub>), 3.14–3.27 m (2H, CH<sub>2</sub>), 3.49–3.54 m (2H, CHSe), 3.70–3.85 m (8H, OCH<sub>2</sub>), 5.94 s (2H, CHO), 5.98–6.04 m (2H, CH<sub>2</sub>CHN), 8.23–8.28 m (2H, H<sub>py</sub>), 8.69–8.74 m (2H, H<sub>py</sub>), 9.08 s (2H, H<sub>py</sub>), 9.15–9.19 m (2H, H<sub>py</sub>). <sup>13</sup>C NMR spectrum,  $\delta_C$ , ppm: 12.7 (CH<sub>3</sub>), 18.4 (CH<sub>2</sub>CH<sub>3</sub>), 25.4 (CH<sub>2</sub>), 28.0

(CH<sub>2</sub>), 28.9 (CHSe), 30.6 (CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>), 67.7 (CH<sub>2</sub>O), 74.6 (CH<sub>2</sub>CHN), 98.4 (HCO), 128.5 (CH<sub>Py</sub>), 140.2 (C<sub>Py</sub>), 140.7 (CH<sub>Py</sub>), 143.2 (CH<sub>Py</sub>), 144.3 (CH<sub>Py</sub>). Found, %: C 53.47; H 6.11; Br 19.34; N 3.56; Se 9.55. C<sub>36</sub>H<sub>48</sub>Br<sub>2</sub>N<sub>2</sub>O<sub>4</sub>Se. Calculated, %: C 53.28; H 5.96; Br 19.69; N 3.45; Se 9.73.

**1,1'-(9-Selenabicyclo[3.3.1]nonane-2,6-diyl)bis[4-(dibutoxymethyl)pyridinium] dibromide (14).** White powder, mp 61–62°C. <sup>1</sup>H NMR spectrum, δ, ppm: 0.89–0.93 m (12H, CH<sub>3</sub>), 1.36–1.45 m (8H, CH<sub>2</sub>CH<sub>3</sub>), 1.62–1.70 m (8H, CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>), 2.43–2.51 m (2H, CH<sub>2</sub>), 2.61–2.71 m (4H, CH<sub>2</sub>), 3.14–3.27 m (2H, CH<sub>2</sub>), 3.48–3.55 m (2H, CHSe), 3.71–3.84 m (8H, OCH<sub>2</sub>), 5.89 s (2H, CHO), 5.95–6.02 m (2H, CH<sub>2</sub>CHN), 8.23 d (4H, H<sub>Py</sub>, *J* = 6.1 Hz), 9.15 d (4H, H<sub>Py</sub>, *J* = 6.1 Hz). <sup>13</sup>C NMR spectrum, δ<sub>C</sub>, ppm: 12.3 (CH<sub>3</sub>), 18.0 (CH<sub>2</sub>CH<sub>3</sub>), 24.9 (CH<sub>2</sub>), 27.5 (CH<sub>2</sub>), 28.7 (CHSe), 30.2 (CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>), 67.7 (CH<sub>2</sub>O), 73.9 (CH<sub>2</sub>CHN), 98.7 (HCO), 125.7 (CH<sub>Py</sub>), 142.9 (CH<sub>Py</sub>), 156.9 (C<sub>Py</sub>). Found, %: C 53.34; H 5.99; Br 19.83; N 3.52; Se 9.98. C<sub>36</sub>H<sub>48</sub>Br<sub>2</sub>N<sub>2</sub>O<sub>4</sub>Se. Calculated, %: C 53.28; H 5.96; Br 19.69; N 3.45; Se 9.73.

The <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on a Bruker DPX-400 spectrometer at 400 and 101 MHz, respectively, using DMSO-*d*<sub>6</sub> as solvent and hexamethyldisiloxane as external standard. Elemental analysis was performed using a Thermo Scientific Flash 2000 automated analyzer. Acetonitrile and methylene chloride were preliminarily dried and distilled. Pyridin-3- and -4-carbaldehydes were commercial products (Sigma–Aldrich).

## CONCLUSIONS

An efficient one-pot procedure has been developed for the synthesis of a new series of water-soluble pyridinium salts from *Z,Z*-cycloocta-1,5-diene, selenium dibromide, and pyridine-3- and -4-carbaldehyde acetals in 80–92% yields. The reactions of 2,6-dibromo-9-selenabicyclo[3.3.1]nonane with 3- and 4-(1,3-dioxolan-2-yl)- and -(1,3-dioxan-2-yl)pyridines gave the corresponding salts in 91–97% yields.

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## CONFLICT OF INTEREST

The authors declare the absence of conflict of interest.

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