SYNTHESIS AND PHYSICO-CHEMICAL CHARACTERIZATION OF SOME QUATERNARY AMMONIUM SALTS OF 2-ARYL THIAZOLE DERIVATIVES

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Abstract
New quaternary ammonium compounds, having at the basis the 2-aryl-thiazole system diversely substituted at the 2, 4, and 5 positions, have been synthesized.

The basic component is represented by tertiary amines in which the nitrogen is included either into an aromatic heterocycle (pyridine, quinoline), or into a tertiary cyclic amine (N-methyl-piperidine, N-methyl-morpholine).

The benzenic ring in the 2 position can be substituted or not. The selected substituents are electron-donating (methyl), or electron-attracting (chlorine).

In order to elucidate the structure of the synthesized compounds, UV, IR, Raman, Mass and ¹H-NMR spectroscopy were performed.

The bactericide activity of the new compounds has been evaluated against two strains of germs: *Staphylococcus aureus* and *Pseudomonas aeruginosa*.

Keywords: quaternary ammonium compounds; 2-aryl-thiazole system; bactericide activity
Introduction

Drugs-resistant germs represent a challenging issue in the anti-infectious therapy. With the view to counteract the increasing prevalence of this phenomenon, a large number of research studies have been focussed on the development of new molecules with antibacterial and/or fungicidal properties [1, 2, 4, 5, 6, 7, 9, 14, 16].

In this context, our research has been focused on the synthesis of new quaternary ammonium salts based on the 2-aryl-thiazole system diversely substituted at the 2, 4, and 5 positions. Moreover, this study was prompted by our previous data which revealed the favourable effects of the thiazole ring associated within the same molecule with a quaternary ammonium group [8, 10, 11, 12, 13, 17].

Materials and methods

The synthesis of these compounds was performed by alkylation of some nitrogen heterocycles, with halogen compounds that have in their structure the 2-aryl-thiazole system. The 2-aryl-halogenomethyl-thiazole derivatives were obtained according to the methods elaborated in our laboratory [10, 15]. We started from diversely substituted thioamides.

In order to enhance the molecular lipophilicity, the compounds substituted with bromine at the position 5 of the thiazole were synthesized.

Chlorine and iodine derivatives (1-5) treated with N-nucleophiles (Nu) in chloroform were transformed into their corresponding ammonium salts (6-17). The basic component is represented by the tertiary nitrogen included into an aromatic (pyridine, quinoline) or non-aromatic heterocycle (N-methyl-pyperidine, N-methyl-morpholine) (Fig. 1):

\[
\begin{align*}
\text{R: } & \text{H, Cl, H}_3\text{C} \\
\text{Nu: } & \text{N-Methyl-pyperidine, N-Methyl-morpholine, N, N-Dimethylpiperazine,} \\
& \text{Pyridine, } 4\text{-Methyl-pyridine, Quinoline} \\
\text{Y: } & \text{H, Br} \\
\text{X: } & \text{Cl, I}
\end{align*}
\]

**Figure 1**
The synthesis of the quaternary ammonium salts
The structures, molecular weights (MW), melting points (Mp) and yields of the obtained compounds are presented in table I.

![Chemical structure](image)

**Table I**

<table>
<thead>
<tr>
<th>Comp. no.</th>
<th>R</th>
<th>Y</th>
<th>X</th>
<th>Nu</th>
<th>MW</th>
<th>Mp °C</th>
<th>Yield</th>
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<tr>
<td>6</td>
<td>H</td>
<td>H</td>
<td>Cl</td>
<td>N-Methyl-pyperidine</td>
<td>308.5</td>
<td>85-86</td>
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<td>7</td>
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<td>I</td>
<td>N-Methyl-pyperidine</td>
<td>400</td>
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<td>8</td>
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<td>228-229</td>
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<tr>
<td>9</td>
<td>Cl</td>
<td>H</td>
<td>I</td>
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<td>434.5</td>
<td>230-232</td>
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<tr>
<td>10</td>
<td>H</td>
<td>H</td>
<td>Cl</td>
<td>N-Methyl-morpholine</td>
<td>310.5</td>
<td>206.7-208</td>
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</tr>
<tr>
<td>11</td>
<td>H</td>
<td>H</td>
<td>I</td>
<td>N-Methyl-morpholine</td>
<td>402</td>
<td>182-185</td>
<td>88%</td>
</tr>
<tr>
<td>12</td>
<td>H</td>
<td>H</td>
<td>I</td>
<td>N,N-dimethyl-pyperazine</td>
<td>716</td>
<td>186-188</td>
<td>48%</td>
</tr>
<tr>
<td>13</td>
<td>H,C</td>
<td>H</td>
<td>I</td>
<td>Pyridine</td>
<td>394</td>
<td>177.2-178.5</td>
<td>40%</td>
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<td>14</td>
<td>Cl</td>
<td>H</td>
<td>I</td>
<td>Pyridine</td>
<td>414.5</td>
<td>187-189</td>
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</tr>
<tr>
<td>15</td>
<td>H</td>
<td>H</td>
<td>I</td>
<td>4-Methyl-pyridine</td>
<td>394</td>
<td>199-200</td>
<td>75%</td>
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<tr>
<td>16</td>
<td>H</td>
<td>Br</td>
<td>I</td>
<td>Quinoline</td>
<td>509</td>
<td>224-226</td>
<td>30%</td>
</tr>
<tr>
<td>17</td>
<td>H,C</td>
<td>H</td>
<td>I</td>
<td>Quinoline</td>
<td>444</td>
<td>198-200</td>
<td>35%</td>
</tr>
</tbody>
</table>

All melting points were determined in open capillaries on Electrothermal IA 9000 apparatus and are uncorrected. The purity of the compounds was routinely checked by thin layer chromatography (TLC) using silica gel with fluorescent indicator 254 nm. Spectroscopic data were recorded on the following instruments: FT-IR/ATR, Bruker Miracle ATR; FT-Raman, Bruker FRA 106/S; 1H-NMR, Bruker Avance – RMN spectrometer in DMSO-d₆ using tetramethylsilane (TMS) as an internal standard, LC-MSD-Trop-VL mass spectrometer; elemental analyses were performed on Elemental Analysen systeme GmbH VarioEL.
Preparation of N-(2-phenyl-thiazole-4-yl-methyl)-N-methyl-pyperidinium chloride (6):
In a flask equipped with a reflux condenser, a mixture of 1 mmol 2-aryl-4-chloromethyl-thiazole and 1 mmol (Nu) was dissolved in 3 ml chloroform. The mixture was heated under reflux for 2 h. The solvent was removed under reduced pressure and the residue was taken again with ether, the solid separated out was filtered and air-dried to give the crude product, which was purified by crystallisation from absolute ethanol to give pure products.

The compounds 7-17 were synthesized in a similar manner.

Results and discussion

The synthesised compounds are white or yellowish-white crystalline powders, soluble in ethanol and in dimethylsulfoxide, less soluble in water, slightly soluble in chloroform and insoluble in ether.

The reactions presented a high yield in case of iodine derivatives, which are more reactive compared to chlorine derivatives. At the same time, the higher nucleophile properties of the N-methyl derivatives induced a higher reaction yield compared to the heteroaromatic tertiary amines.

To elucidate the structures of the resulting compounds the elemental analysis, as well as UV, IR, Raman, MS and 1H-NMR were performed.

The UV spectra of these compounds present a maximum absorption between 283 nm and 284.5 nm (which is assigned to the benzene ring), shifting towards higher wavelengths (289 nm to 290 nm) when the benzene ring in the 2 position of thiazole is substituted with methyl or chlorine. If the 5 position of the thiazole ring is substituted with bromine, the maximum absorption becomes visible at 293.5 nm.

The Raman spectra, recorded for the synthesized compounds, revealed the presence of N=C endocyclic bonds between 1680-1500 cm⁻¹, the valence vibrations of aromatic C-H bonds are present at the 3100-3000 cm⁻¹, and the valence vibrations corresponding to the aliphatic C-H are present between 2990-2800 cm⁻¹.

As an example, the Raman spectra of N-(2-phenyl-thiazole-4-yl-methyl)-N-methyl-pyperidinium iodide is presented. In Raman spectra, an intense band was observed at 1600 cm⁻¹ attributed to the N=C endocyclic bond in the thiazole system; the bands between 3017 cm⁻¹ and 3074 cm⁻¹ were attributed to valence vibration induced by aromatic C-H, whilst the bands between 2977 cm⁻¹ and 2869 cm⁻¹ were ascribed to the aliphatic C-H (Fig. 2).
The $^1$H-NMR spectra were performed in liquid samples using deuterated dimethylsulphoxide (DMSO). The following signals have been evidenced in the $^1$H-NMR spectrum of N-(2-phenyl-thiazole-4-yl-methyl)-N-methyl pyperidinium chloride (Fig. 3), and the attribution of the signals is presented in table II.
Table II
The attribution of the signals
for N-(2-phenyl-thiazole-4-yl-methyl)-N-methyl pyperidinium chloride

<table>
<thead>
<tr>
<th>2H</th>
<th>2H</th>
<th>2H</th>
<th>DMSO</th>
<th>3H</th>
<th>4H (8,12)</th>
<th>2H (7)</th>
<th>2H (2,3,4)</th>
<th>2H (1,5)</th>
<th>1H</th>
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<tr>
<td>1.572</td>
<td>1.831</td>
<td>1.952</td>
<td>2.508</td>
<td>3.096</td>
<td>4.786</td>
<td>7.538</td>
<td>7.972</td>
<td>8.135</td>
<td></td>
</tr>
<tr>
<td>1.585</td>
<td>1.851</td>
<td>1.965</td>
<td>3.74</td>
<td>3.434</td>
<td>3.443</td>
<td>7.544</td>
<td>7.977</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.600</td>
<td>1.859</td>
<td>3.395</td>
<td>3.420</td>
<td>3.454</td>
<td>7.553</td>
<td>7.986</td>
<td></td>
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<td></td>
</tr>
</tbody>
</table>

The recorded mass spectra have the molecular peaks in accordance to the synthesized compounds. In the mass spectra of 2-(4-methyl-phenyl)-thiazol-4-yl-methyl-quinolinium iodide the molecular peak of the compound without iodide, m/z 317, was obtained. Besides this, other peaks specific to the heterocyclic fragmentation were presented (m/z 188 and 129). With a relative high abundance, the peaks characteristic for the fragmentation of the 2-aryl-thiazolic (m/z 118, 91, 135, 71, 45) were observed (Fig. 4).

![Figure 4](image_url)

MS fragmentation of 2-(4-Methyl-phenyl)-thiazol-4-yl-methyl-quinolinium iodide

The obtained physico-chemical data provide strong evidences, which are in accordance with the attributed structures.
N-(2-phenyl-thiazole-4-yl-methyl)-N-methyl pyperidinium chloride (iodide) (6, 7)

\[
\begin{array}{c}
\text{H}_3C \\
\text{N} \\
\text{S} \\
\text{C} \\
\text{N} \\
\text{C} \\
\text{CH}_2 \\
\text{N} \\
\text{C} \\
\text{Y} \\
\text{X} \\
\end{array}
\]

\(^1\)HNMR (500 MHz, DMSO-\(d_6\)) \(\delta\) (ppm): 1.57-1.60 (2H, t, C\(_{10}\) pyperidine), 1.83-1.86 (2H, t, C\(_{11}\) pyperidine), 1.95-1.96 (2H, d, C\(_9\) pyperidine), 3.09 (3H, s, CH\(_3\)), 3.44-3.46 (4H, 2d, C\(_{8,12}\) pyperidine), 4.78 (2H, s, C\(_7\) methylene), 7.53-7.55 (3H, t, C\(_{2,3,4}\) phenyl), 7.97-7.99 (2H, 2d, C\(_{1,5}\) phenyl), 8.13 (1H, s, C\(_6\) thiazole);

For C\(_{16}H\(_{21}\)ClN\(_2\)S calculated: 62.24% C, 6.81% H, 9.08% N, 10.37% S; found 62.28% C, 6.80% H, 9.04% N, 10.39% S; MS: m/z: 273 [M-MCl].

N-(5-Bromo-2-phenyl-thiazole-4-yl-methyl)-N-methyl-pyperidinium iodide (8)

\(^1\)HNMR (500 MHz, DMSO-\(d_6\)) \(\delta\) (ppm): 1.59-1.60 (2H, t, C\(_{10}\) pyperidine), 1.88-1.89 (2H, t, C\(_{11}\) pyperidine), 1.94-1.95 (2H, d, C\(_9\) pyperidine), 3.11 (3H, s, CH\(_3\)), 3.55-3.57 (4H, 2d, C\(_{8,12}\) pyperidine), 4.68 (2H, s, C\(_7\) methylene), 7.55-7.57 (3H, q, C\(_{2,3,4}\) phenyl), 7.93-7.95 (2H, 2d, C\(_{1,5}\) phenyl);

For C\(_{16}H\(_{20}\)BrIN\(_2\)S calculated: 40.08% C, 4.18% H, 5.85% N 6.68% S; found 40.00% C, 4.20% H, 5.95% N, 6.65% S; MS: m/z: 352 [M-MI]; Raman \(\nu_{\text{max}}\) (cm\(^{-1}\)): 1600 (N=C endocyclic), 2863-2918 (C-H aliphatic), 3005-3012 (C=H aromatic).

N-2-(4-Cloro-phenyl)-thiazole-4-yl-methyl-N-methyl-pyperidinium iodide (9)

\(^1\)HNMR (500 MHz, DMSO-\(d_6\)) \(\delta\) (ppm): 1.57-1.60 (2H, t, C\(_{10}\) pyperidine), 1.83-1.86 (2H, t, C\(_{11}\) pyperidine), 1.95-1.96 (2H, d, C\(_9\) pyperidine), 3.09 (3H, s, CH\(_3\)), 3.44-3.46 (4H, t, C\(_{8,12}\) pyperidine), 6.05 (2H, s, C\(_7\) methylene), 7.77-7.96 (2H, 2d, C\(_{1,5}\) phenyl), 7.32-7.55 (2H, t, C\(_{2,4}\) phenyl), 7.97 (1H, s, C\(_6\) thiazole);

For C\(_{16}H\(_{20}\)ClIN\(_2\)S calculated: 44.19% C, 4.60% H, 6.44% N, 7.36% S; found 44.25% C, 4.53% H, 6.60% N, 7.25% S; MS: m/z: 307.5[M-MI]; Raman \(\nu_{\text{max}}\) (cm\(^{-1}\)): 1602 (N=C endocyclic), 2863-2918 (C-H aliphatic), 3050-3075 (C=H aromatic).
N-2-Phenyl-thiazole-4-yl-methyl-N-methyl-morpholinium chloride (iodide) (10, 11)

1H NMR (500 MHz, DMSO-d_6) δ (ppm): 3.21 (3H, s, CH_3), 3.46-3.52 (2H, d, C_{11} morpholine), 3.64-3.71 (2H, t, C_9 morpholine), 3.99-4.01 (2H, t, C_{12} morpholine), 4.08-4.05 (2H, d, C_8 morpholine), 5.00 (2H, s, C_7 methylene), 7.53-7.55 (3H, t, C_{2,3,4} phenyl), 7.97-7.99 (2H, 2d, C_{1,5} phenyl), 8.22 (1H, s, C_6 thiazole);

For C_{15}H_{19}ClO_N_2S calculated: 57.97% C, 6.12% H, 9.02% N, 10.31% S; found 57.89% C, 6.14% H, 9.10% N, 10.30% S. m/z: 275 [M-MCl].

For C_{15}H_{19}I_2N_2O_S calculated: 44.78% C, 4.73% H, 6.97% N, 7.96% S; found 44.79% C, 4.70% H, 6.94% N, 7.93% S; MS: m/z: 275 [M-MI]; Ramman ν_{max} (cm⁻¹): 1596 (N=C endocyclic), 2950-2963 (C-H aliphatic), 3052-3059 (C=H aromatic).

N,N-(2-Phenyl-thiazole-4-yl-methyl)-N,N-dimethyl-pyperazinium iodide (12)

1H NMR (500 MHz, DMSO-d_6) δ (ppm): 2.31 (6H, s, 2 CH_3), 2.65-2.72 (2H, t, C_{12} pyperazine), 2.85-2.9 (2H, d, C_{10} pyperazine), 3.38-3.50 (2H, t, C_9 pyperazine), 3.49-3.60 (2H, t, C_8 pyperazine), 4.83 (4H, s, C_7 methylene), 7.55-7.57 (6H, t, C_{2,3,4} phenyl), 7.97-8.00 (4H, 2d, C_{1,5} phenyl), 8.13 (2H, s, C_6 thiazole);

For C_{26}H_{30}I_2N_4S_2 calculated: 43.58% C, 4.19% H, 7.82% N, 8.94% S; found 43.71% C, 4.02% H, 7.62% N, 9.00% S; MS: m/z: 462 [M-M_{2I}]; Ramman ν_{max} (cm⁻¹): 1595 (N=C endocyclic), 2939-2970 (C-H aliphatic), 3026-3059 (C=H aromatic).

2-(4-Methyl)-phenyl-thiazole-4-yl-methyl-pyridinium iodide (13)

1H NMR (500 MHz, DMSO-d_6) δ (ppm): 2.34 (3H, s, CH_3), 6.02 (2H, s, C_7 methylene), 7.31-7.32 (2H, d, C_{2,4} phenyl), 7.77-7.79 (2H, 2d, C_{1,5} phenyl), 7.97 (H, s, C_6 thiazole), 8.20-8.24 (2H, t, C_{9,11} pyridine), 8.64-8.68 (H, t, C_{10} pyridine) 9.23-9.25 (2H, d, C_8, C_{12} pyridine);

For C_{18}H_{15}I_N_2S calculated: C 48.73%, H, 3.81% N, 7.11% S 8.12%; found 48.70% C, 3.84% H, 7.08% N, 8.09% S; MS: m/z: 267 [M-MI]; Ramman ν_{max} (cm⁻¹): 1600 (N=C endocyclic), 3050-3080 (C=H aromatic).

2-(4-Cloro)-phenyl-thiazole-4-yl-methyl-pyridinium iodide (14)

1H NMR (500 MHz, DMSO-d_6) δ (ppm): 6.02 (2H, s, C_7 methylene), 7.30-7.32 (2H, d, C_{2,4} phenyl), 7.77-7.79 (2H, d, C_{1,5} phenyl), 7.97 (H, s, C_6 thiazole), 8.20-8.24 (2H, t, C_8, C_{11} pyridine), 8.47-8.68 (H, t, C_{10} pyridine), 9.23-9.25 (2H, d, C_8, C_{12} pyridine),
For C_{15}H_{12}ClIN_{2}S calculated: 43.42% C, 2.89% H, 6.76% N, 7.72% S; found 43.50% C, 2.91% H, 6.60% N, 7.75% S; MS: m/z: 287.50 [M-M_{I}]; Ramman $\nu_{\text{max}}$ (cm$^{-1}$): 1602 (N=C endocyclic), 3020-3065 (C=H aromatic).

2-Phenyl-thiazole-4-yl-methyl-(4-methyl)-pyridinium iodide (15)
$^1$HNMR (500 MHz, DMSO-d$_6$) $\delta$ (ppm): 2.62 (3H, s, CH$_3$), 5.97 (2H, s, C$_7$ methylene), 7.50-7.51 (3H, t, C$_{2,3,4}$ phenyl), 7.89-7.90 (2H, 2d, C$_{1,5}$ phenyl), 8.01 (H, s, C$_6$ thiazole), 8.04-8.03 (2H, d, C$_9$, C$_{11}$ pyridine), 9.10-9.09 (2H, d, C$_8$, C$_{12}$ pyridine);
For C$_{16}$H$_{15}$IN$_2$S calculated: 48.73% C, 3.81% H, 7.11% N, 8.12% S; found 48.75% C, 3.79% H, 7.13% N, 8.10% S; MS: m/z: 267 [M-M$_I$], Ramman $\nu_{\text{max}}$ (cm$^{-1}$): 1598 (N=C endocyclic), 3000-3070 (C=H aromatic).

5-Bromo-2-phenyl-thiazole-4-yl-methyl-quinolinium iodide (16)
$^1$HNMR (500 MHz, DMSO-d$_6$) $\delta$ (ppm): 6.40 (2H, s, C$_7$ methylene), 7.47-7.45 (3H, 2d, C$_{2,3,4}$ phenyl), 7.80 -7.78 (2H, 2d, protons C$_{1,5}$ phenyl), 8.04-8.07 (1H, t, proton C$_9$ quinoline), 8.30-8.27 (1H, t, roton C$_{12}$ quinoline), 8.35-8.32 (1H, 2d, C$_{13}$ quinoline), 8.52-8.51 (1H, d, C$_{11}$ quinoline), 8.81-8.79 (1H, d, C$_8$ quinoline), 9.40-9.38 (1H, d, C$_{10}$ quinoline), 9.86-9.85 (1H, d, C$_{14}$ quinoline);
For C$_{19}$H$_{14}$BrIN$_2$S calculated: 44.79% C, 2.75% H, 5.50% N, 6.29% S; found 44.85% C, 2.82% H, 5.60% N, 6.31% S; MS: m/z: 382 [M-M$_I$], Ramman $\nu_{\text{max}}$ (cm$^{-1}$): 1631 (N=C endocyclic), 3000-3025 (C=H aromatic).

2-(4-Methyl)-phenyl-thiazole-4-yl-methyl-quinolinium iodide (17)
$^1$HNMR (500 MHz, DMSO-d$_6$) $\delta$ (ppm): 2.3 (3H, s, CH$_3$), 6.40 (2H, s, C$_7$ methylene), 7.25-7.27 (2H, d, C$_{2,4}$ phenyl), 7.67 -7.69 (2H, 2d, C$_{1,5}$ phenyl), 8.03-8.07 (1H, 2d, C$_{13}$ quinoline), 8.06 (1H, s, C$_6$ thiazole), 8.27-8.31 (1H, t, C$_9$ quinoline), 8.32-8.34 (1H, t, C$_{12}$ quinoline), 8.50-8.52 (1H, d, C$_{11}$ quinoline), 8.77-8.79 (1H, d, C$_8$ quinoline), 9.37-9.39 (1H, d, C$_{10}$ quinoline), 9.81-9.82 (1H, d, C$_{14}$ quinoline);
For C$_{20}$H$_{17}$IN$_2$S calculated: 54.05% C, 3.83% H, 6.31% N, 7.21 S; found 54.25% C, 3.66% H, 6.48% N, 7.03% S; MS: m/z: 317[M-M$_I$]; Ramman $\nu_{\text{max}}$ (cm$^{-1}$): 1625 (N=C endocyclic), 3025-3075 (C=H aromatic).
Some compounds were assessed for their bactericide activity on *Pseudomonas aeruginosa* ATCC15442 and *Staphylococcus aureus* ATCC6538. In order to be considered a molecule with bactericidal activity, these compounds have to demonstrate at least a 5 decimal log reduction of at least two test microorganisms *Pseudomonas aeruginosa* ATCC 15442 (1) and *Staphylococcus aureus* ATCC 6538 (2), at 20°C, after 5 minutes contact time, if the basic limits of the method are accomplished [3, 17, 18, 19, 20]. Initially, the number of the germs was 4x10⁸ for *Pseudomonas aeruginosa* and 8x10⁸ for *Staphylococcus aureus*. The obtained data are presented in table III. Only the compounds 8, 11 and 16 have presented a low bactericide activity.

### Table III

<table>
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<th>No. comp.</th>
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<th>(2)</th>
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<td>countless</td>
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<tr>
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<td>&lt;4000</td>
<td>&lt;3500</td>
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<tr>
<td>11</td>
<td>&lt;4000</td>
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<tr>
<td>16</td>
<td>&lt;4000</td>
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### Conclusions

New quaternary ammonium compounds that have in their structure the 2-aryl-thiazole system were synthesized.

In order to ascertain the structure of resulting compounds IR, Raman, MS and ¹H-NMR spectroscopy were performed.

Some compounds were assessed for their bactericide action on *Pseudomonas aeruginosa* and *Staphylococcus aureus*. The compounds 8, 11 and 16 have presented a low bactericide action.

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### References

19. ***SR EN 1040: 2006, 5.5.2 Dilution-neutralization method, 12

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