Mono-substituted p-tert-butylthiacalix[4]arene: Regioselective Synthesis and Toxicological Activity Against Cowpea Aphid, Aphis craccivora (Koch)

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Received: 11th April 2024, Revised: 29th June 2024, Accepted: 18th July 2024
Published online: 24th July 2024

Abstract: Tetraethylammonium bromide (TEAB) in dry benzene has been utilized as the base enabling Phase Transfer Catalysis PTC for the synthesis of some novel mono-substituted thiacalix[4]arene 2 and 3 at the lower rim from 2-chloro(p-tolyl)acetamide and 2-chloro(p-acetylphenyl)acetamide, respectively. Bromine and mono-p-acetylphenylacetamide-p-tert-butylthiacalix[4]arene 3 were reacted in chloroform under solar radiation to give mono-p-phenacylbromideacetamide-p-tert-butylthiacalix[4]arene 4. Derivatives of mono-substituted thiacalix[4]arenes 2-4 were reacted with Lawesson reagent (LR) affording mono-substituted thiacalix[4]arene derivatives 5-7, respectively. The temple of the novel compounds was illustrated based on elemental analyses, 1H-, 13C-, and Dept-NMR spectroscopy. Synthesized derivatives have been tested against adult and nymph Cowpea Aphid (Aphis craccivora) in laboratory conditions as insecticidal agents, to calculate the LC50 values for each one of them. Bioassay experiments showed that synthesized compounds 4 and 7 possess the highest insecticidal bio-efficacy, with LC50 values of 1.16 and 2.08 ppm, respectively against nymphs, and 11.96 and 13.52 ppm, respectively, against adults of Aphis craccivora.

Keywords: Thiacalix[4]arene, Tetraethylammonium bromide, Lawesson reagent, Aphis craccivora, Eco-friendly insecticides.

1. Introduction

The original synthesis of p-tert-butylthiacalix[4]arene (TCA) substituted sulfur atom bridges for methylene groups that linked the phenolic moieties was reported by Miyano et al. in 1997. [1]. This class of macrocycles has become more popular due to its ease of preparation and wide range of applications [2-7]. TCA has the ability for selective alkylation due to containing four hydroxyl groups. Initial research on the chemistry of thiacalix[4]arenes primarily involved adjustments at the low rim[6,7]. The conventional Williamson method to synthesize ethers, which involves alkyl halides and a base, is used to derivatize thiacalixarenes and make similar alterations [8]. According to the circumstances and the proportion of thiacalixarenes, alkylation reagents, and base, their reactions gave 1,3-di or tetrasubstituted items [9]. Dialkylation of thiacalixarenes led ordinarily to distally (1,3- ) di-substituted items [8-12]. On the other hand, the upper rim substituted thiacalix[4]arenes are active sites other than the lower rim [11]. In 2004, according to Kon et al., ethyl bromoacetate was alkylated under nitrogen gas conditions with TCA in a regulated manner selectively for the synthesis of monosubstituted p-tert-butylthiacalix[4]arene [13]. This approach opened several avenues for the manufacture of monosubstituted p-tert-butylthiacalix[4]arene derivatives. Recently, researchers have begun to develop new methods to produce these products. For example, Lamouchi et al. in 2012 synthesized the monosubstituted p-tert-butylthiocoly[4]arene in two steps, through which the substituted TCA was first precipitated via a phase transition catalyst, using tetrabutylammonium bromide and dimethylformamide [14]. Also, Omran in 2016 succeeded in synthesis of monosubstituted p-tert-butylthiacalix[4]arene under Phase Transfer Catalysis PTC, which is faster than the latter method [15]. On the other hand, calixarenes are known as safe materials and are used to get rid of pesticide residues [16-18]. Also, their derivatives have toxicity against bacteria and fungi [19].

Here in this work, we aim to synthesize some new derivatives of p-tert-butylthiacalix[4]arene at the lower rim and in addition to eco-friendly insecticides against aphids, which causes huge damage to many crops around the world.

2. Results and Discussion

Phase transfer catalysis was found to be the most suitable technique for the synthesis of monosubstituted p-tert-butylthiocalix[4]arenes. Thus, the reaction of p-tert-butylthiocalix[4]arene 1 with mole equivalent of 2-chloro-N-(p-tolyl)acetamide and/or 2-chloro(p-acetyl phenyl)acetamide using potassium iodide, potassium carbonate and tetraethylammonium bromide (TEAB) in dry benzene gave mono substituted p-tert-butylthiocalix[4]arene 2 and 3, respectively, in an excellent yield. (Scheme 1).
The reaction condition for the synthesis of mono-thiacalix[4]-arenes 3 was optimized using different solvent; base and reaction time to improve the yield of 5,11,17,23-tetra-tert-butyl-25-[N-(4-acetylenyl)-acetamide]-26,27,28-trihydroxy-thiacalix[4]arene 3. Table 1. From these data, we found that the yield was dependent on (i) Base strength; (ii) Solvent type; and (iii) Time of the reaction.

### Table 1: The optimal circumstances for the synthesis of TCA 3.

<table>
<thead>
<tr>
<th>Base</th>
<th>Solvent</th>
<th>Time (h)</th>
<th>Yield (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Na₂CO₃</td>
<td>Benzene</td>
<td>30</td>
<td>62</td>
</tr>
<tr>
<td>Na₂CO₃</td>
<td>Acetone</td>
<td>48</td>
<td>43</td>
</tr>
<tr>
<td>Na₂CO₃</td>
<td>Dioxane</td>
<td>48</td>
<td>34</td>
</tr>
<tr>
<td>Na₂CO₃</td>
<td>Dioxane</td>
<td>36</td>
<td>23</td>
</tr>
<tr>
<td>K₂CO₃</td>
<td>Benzene</td>
<td>3</td>
<td>89*</td>
</tr>
<tr>
<td>K₂CO₃</td>
<td>Acetone</td>
<td>36</td>
<td>54</td>
</tr>
<tr>
<td>K₂CO₃</td>
<td>Dioxane</td>
<td>24</td>
<td>60</td>
</tr>
<tr>
<td>K₂CO₃</td>
<td>Dioxane</td>
<td>24</td>
<td>36</td>
</tr>
</tbody>
</table>

It seems that the non-polar character of benzene solvent and the reaction temperature (≥ or = 80 °C) accelerate the alkylation of Calixarenes OH and lowering the self alkylation of acetamides (dimerization) through COCH₃ in one molecule and COCH₂₃ in another molecule: which is clear in other used solvents.

The spectral data (IR, ¹H NMR, ¹³C NMR, and Dept-NMR) of the newly synthesized compounds confirmed their chemical structure. (see Experimental Part & SI). The infrared spectra of mono-p-tolylacetamide-p-tert-butylthiacalix[4]arene 2 showed new characteristic absorption bands for amino N-H and two carbonyl C=O groups at δ 3337 cm⁻¹ and carbonyl C=O at δ 1686 cm⁻¹. Its ¹H NMR spectrum showed the appearance of two singlet signals at δ 1.24 & 1.29 ppm for Bu protons; two singlet signals at δ 2.38 & 4.87 ppm for methyl and methylene protons, respectively; multiplet signals at δ 7.24-7.82 ppm for aromatic protons, three OH protons appeared as two singlet signals at δ 9.24 and 9.52 ppm; and broad singlet signal at δ 10.63 ppm for NH proton. The ¹³C NMR spectrum revealed seventeen signals in the aromatic region for sp²-carbons at δ 120.0, 120.6, 120.7, 128.2, 129.5, 133.9, 135.6, 136.0, 136.4, 137.0, 144.4, 144.6, 150.4, 155.8, 156.3, 157.4, and 165.8 ppm; and six signals in the aliphatic region for p-tert butyl and O-CH₂ group carbons at δ 20.9, 31.0, 31.3, 34.3, 34.6, and 76.4 ppm, respectively. The infrared spectrum of mono-p-acetylenylacetamide-p-tert-butylthiacalix[4]arene 3 showed new absorption bands for amino N-H and two carbonyl C=O groups at δ 3061, 1789, and 1682, respectively. The ¹H NMR spectrum of compound 3 confirmed the appearance of Bu¹ protons as two singlet signals at δ 1.13 and 1.17 ppm; two singlet signals at δ 2.53 and 4.77 ppm for methyl and methylene protons, respectively; multiple signals in the aromatic region at δ 7.18-7.95 ppm for aromatic protons; two singlet signals at δ 9.18 and 9.46 ppm due to three protons of hydroxyl group; and singlet signal at δ 10.87 ppm for NH proton. The ¹³C NMR spectrum of compound 3 demonstrated twenty signals at δ 119.8, 120.0, 120.6, 124.2, 128.1, 128.3, 129.7, 133.2, 136.0, 136.4, 137.0, 142.4, 144.6, 144.7, 150.8, 155.7, 156.1, 157.2, 166.4, and 197.0 ppm due to carbons of aromatic and carbonyl groups; and seven signals in the aliphatic region at δ 26.4, 30.9, 31.3, 34.2, 34.3, 34.6, and 76.2 (exchangeable by Dept NMR) ppm assigned to carbons of p-tert butyl group, CH₃, and O-CH₂, respectively. The reaction of mono-p-acetylenylacetamide-p-tert-butylthiacalix[4]arene 3 with one equivalent mole of bromine in chloroform under solar radiation (sunlight) produced 5,11,17,23-tetra-tert-butyl-25-[N-(4-(2-bromoacetyl)phenyl)-acetamide]-26,27,28-trihydroxy-thiacalix[4]arene 4. (Scheme 2).

The infrared spectrum of compound 4 presented the emergence of distinctive absorption bands at δ 3491, 3380, 3060, and 2906 cm⁻¹ for OH, NH, CH₃am. and CH₃aliph. groups, respectively; 1785 cm⁻¹ for the carbonyl ketone group; and 1678 cm⁻¹ for the carbonyl group. Its ¹H NMR spectrum revealed that tert-butyl protons appeared as two singlet signals at δ 1.23 ppm for nine protons and at δ 1.28 ppm for twenty-seven protons; two singlet signals at δ 4.48 and 4.88 ppm due to protons of (O-CH₂) and (Br-CH₂), respectively; multiple signals at δ 7.68 8.10 ppm for aromatic protons; while three protons of hydroxyl phenolic groups appeared as two singlet signals at δ 9.25 and 9.53 ppm; and protons of NH appeared as an individual signal at δ 10.98 ppm. Its ¹³C NMR spectrum presented seventeen signals at δ 120.0, 120.6, 128.1, 129.8,
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130.0, 136.0, 136.4, 143.3, 144.6, 144.8, 150.8, 155.7, 156.1, 157.2, 166.6, and 190.1 ppm for sp²-carbons; seven signals in aliphatic region at δ 30.8, 31.0, 31.3, 34.2, 34.3, 34.6 and 76.2 ppm for aliphatic carbons. Its dept-135 showed the appearance of two signals for two methylene carbons of (O-CH₂) and (Br-CH₂), in the opposite direction at δ 30.8 and 76.2 ppm, respectively three signals for CH aliphatic carbon at δ 31.0, 31.3, and 31.3 ppm; and five signals in the aromatic region at δ 120.0, 136.0, 136.4, 136.5 and 137.1 ppm due to the CH aromatic carbons.


![Scheme 3](image)

Scheme 3. Synthesis of compounds 5-7

Infrared in the mono-p-tolylthioacetamide-p-tert-butyllthialcalix[4]arene 5 spectrum, the distinctive carbonyl (C=O) group absorption band vanished. Its ¹H NMR spectrum showed the appearance of two singlet signals for four tert-butyl protons at δ 1.24 and 1.29 ppm; two singlet signals for methyl and methylene protons at δ 2.42 and 5.26 ppm, respectively; two singlet signals for sp²-aromatic protons in the aromatic region at δ 7.32–7.98 ppm; two singlet signals for three OH protons at δ 9.18 and 9.51 ppm; and singlet signal for NH proton at δ 12.22 ppm.

3.1 Insecticidal bio-efficacy screening:
Calixarene compounds have many uses and applications especially separation [16], purification [17], and pollution control [18]. Thus, calixarenes are classified as eco-friendly compounds, however, the last year’s toxicity of calixarenes against bacteria and fungi was recorded [19], here a novel attempt to demonstrate the insecticidal efficacy of calixarene compounds against Cowpea aphids, with the hope to find out eco-friendly insecticides, avoiding the traditional chemical insecticides with their harmful impact to the environment.

To determine the LC50 values, each toxicological data about aphid mortality was examined using probity analysis using a statistical program (LDP-line). The results are shown in Table 2 and Figure 1, including slope, toxic ratio, and LC50 values.

3.2. A) Toxicological activity against nymphs of Aphis craccivora after 24 h of treatment.

The results, which are displayed in Table 2, demonstrate that all six tested substances exhibit weak to moderate toxicological action against A. craccivora nymphs, with LC50 values ranging from 1.16 to 6.54 ppm during a 24-hour treatment period. In particular, LC50 values of compounds 2, 3, 4, 5, 6, and 7 are 6.26, 6.54, 1.16, 3.91, 5.29, and 2.08 ppm respectively. Compounds 4 and 7 had the highest toxicological activity of all the investigated derivatives, with LC50 values of 1.16 and 2.08 ppm, respectively; due to the presence of Br atom in the molecules.

Table 2. Insecticidal activity of compounds 2, 3, 4, 5, 6, and 7 after 24 h of treatment against nymphs and adults of Aphis craccivora insects

<table>
<thead>
<tr>
<th>Comp.</th>
<th>LC50 (ppm)</th>
<th>Slope</th>
<th>Toxic ratio</th>
<th>LC50 (ppm)</th>
<th>Slope</th>
<th>Toxic ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>15.03</td>
<td>1.0388 ± 0.4334</td>
<td>0.79</td>
<td>6.26</td>
<td>0.9772 ± 0.3876</td>
<td>0.18</td>
</tr>
<tr>
<td>3</td>
<td>13.62</td>
<td>0.8147 ± 0.3737</td>
<td>0.87</td>
<td>6.54</td>
<td>0.6066 ± 0.2349</td>
<td>0.17</td>
</tr>
<tr>
<td>4</td>
<td>11.96</td>
<td>0.6629 ± 0.3781</td>
<td>1</td>
<td>1.16</td>
<td>0.3424 ± 0.2189</td>
<td>1</td>
</tr>
<tr>
<td>5</td>
<td>18.71</td>
<td>0.8634 ± 0.3772</td>
<td>0.64</td>
<td>3.91</td>
<td>0.4715 ± 0.2271</td>
<td>0.29</td>
</tr>
<tr>
<td>6</td>
<td>22.00</td>
<td>0.9571 ± 0.3883</td>
<td>0.54</td>
<td>5.29</td>
<td>0.5620 ± 0.2320</td>
<td>0.22</td>
</tr>
<tr>
<td>7</td>
<td>13.52</td>
<td>0.6359 ± 0.3773</td>
<td>0.88</td>
<td>2.08</td>
<td>0.3944 ± 0.2221</td>
<td>0.55</td>
</tr>
</tbody>
</table>

![Fig. 1](image)

Fig. 1: Insecticidal activity of compounds 2, 3, 4, 5, 6, and 7 against nymphs and adults of Aphis craccivora insects after 24 hrs of treatment.

3.2. B) toxicological activity against Aphis craccivora adults over a 24-hour treatment period.

According to the insecticidal activities of derivatives 2, 3, 4, 5, 6, and 7 against adults of A. craccivora insect, after 24 hrs of
treatment, all chemicals exhibited LC50 values ranging from 11.96 to 22.00 ppm, indicating moderate to mild toxicological action against adult A. craccivora. With LC50 values of 11.96 and 13.52, respectively, the analogs 4 and 7 exhibited the highest levels of toxicological activity. Compounds 2, 3, 4, 5, 6, and 7 have LC50 values of 15.03, 13.62, 11.96, 18.71, 22.00, and 13.52 ppm, respectively.

4. Experimental

4.1. Chemistry

**General Information:**

We used all commercially available reagents without further purification; they were acquired from Fluka, Aldrich, and Merck. For all reactions, thin-layer chromatography (TLC) was employed. Percolated silica gel G/UV-254 plates with a 0.25 mm thickness (Merck 60F254) were used, and UV light (254 nm/365 nm) was used for visualization. A Kofer melting points instrument was used to find the uncorrected melting points. An FT-IR-ALPHABROKER-Platinum-ATR spectrometer was used to gather infrared spectra using the attenuated total reflection (ATR) method. The spectra are displayed as cm⁻¹.

The ¹H and ¹³C NMR spectra of each chemical were acquired with a Bruker Bio Spin AG spectrometer running at 400 MHz and 100 MHz, respectively, in DMSO-d₆ and CDCl₃. The water TQD quadruple spectrometer, which is connected to the chemical were acquired with a Perkin-Elmer CHN-analyzer model.


A mixture of TCA 1 (1g, 1.38 mmole), 5 gm of anhydrous K₂CO₃, 0.5 gm TEAB, potassium iodide (2.7 mmole), and 2-chloro-N-(p-acetylphenyl)acetamide (0.33g, 1.56 mmole) refluxed in 20 mL of benzene while stirring for 3 hrs. After filtering out the reaction mixture, the benzene layer was dried out by evaporating it under low pressure. The solid residue was washed with hydrochloric acid and then extracted with CH₂Cl₂. Next, methanol was added to the layer of saturated CH₂Cl₂. The resulting white solid was dried and filtered out. Methanol was added after the concentration of the CH₂Cl₂ layer. Filtered off and allowed to dry, the white solid developed. (3, C₅H₆NO₂S₂) Yield 89%; white solid, mp 160 °C; MS: m/z 895.31 [MH]+; IR (KBr): νmax 3386 (OH) 3337 (NH) 3061 (COₐromatic), 1785 (COₐromatic), 1678 (COamide) cm⁻¹; ¹H NMR (CDCl₃) δ (ppm): 1.13 (9H, s, Bu'), 1.17 (27H, s, Bu), 2.53 (3H, s, CH₃), 4.77 (2H, s, CH₂), 7.18 (1H, br. s, Ar-H), 7.57-7.65 (9H, m, Ar-H), 7.95 (4H. br. s, Ar-H), 9.18 (2H, Ar-H), 9.46 (1H, s, OH), 10.87 (1H, s, NH); ¹³C NMR (CDCl₃) δ (ppm): 26.4, 30.9, 31.3, 34.2, 34.3, 34.6, 76.2(exchangeable), 119.8, 120.0, 120.6, 124.2, 128.1, 128.3, 129.7, 133.2, 136.0, 136.4, 137.0, 142.4, 144.4, 144.7, 150.8, 155.7, 156.1, 157.2, 166.4, 197.0.


To a solution of p-(acetylphenyl)acetamide-TCA 3 (1g, 1.11 mmole) in 15 mL of dry chloroform. After adding the bromine (0.059 mL, 0.36 mmole) dropwise and stirring the reaction liquid for five minutes in the presence of sunlight, the reaction was allowed to continue. After that, the reaction mixture was then filtered for thirty minutes. Product 4 was separated after the addition of 5 mL of methanol into the reaction mixture and recrystallized from a mixture of methanol: chloroform (1:3). (4, C₅H₅BrNO₂S) Yield 91%; white solid, mp 225 °C; MS: m/z 893.29 [MH]+; IR (KBr): νmax 3349 (OH), 3380 (NH), 3060 (CHₐromatic), 2906 (CHₐliph), 1785 (COₐromatic), 1678 (COₐromatic), 1785 (COₐromatic), 1678 (COamide) cm⁻¹; ¹H NMR (CDCl₃) δ (ppm): 1.23 (9H, s, Bu'), 1.28 (27H, s, Bu), 4.48 (2H. s, CH₂), 4.88 (2H, s, CH₂), 7.68-7.75 (8H, m, CHₐromatic), 8.05-8.10 (4H, m, CHₐromatic), 9.25 (2H, s, OH), 9.53 (1H, s, OH), 10.98 (1H, s, NH); ¹³C NMR (CDCl₃) δ (ppm): 30.8 (exchangeable), 31.0, 31.3, 34.2, 34.3, 34.6, 76.2(exchangeable), 120.0, 120.6, 128.1, 129.8, 130.0, 136.0, 136.4, 137.1, 143.3, 144.6, 144.8, 150.8, 158.0, 158.7, 156.1, 157.2, 166.6, 190.1.


A combination of Lawesson's reagent (0.23 g 0.57 mmole) and mono-p-tolylacetamide-TCA 2 (1g, 1.15 mmole) refluxed in 20 mL of benzene while stirring for 3 hrs. A whitish-yellow solid product was precipitated, filtered, and crystallized by a mixture of methylene chloride and methanol (2:1). (5, C₅H₆NO₂S₂) Yield 63%; Whitish yellow crystals, mp 240 °C; MS: m/z 883.29 [MH]+; IR (KBr): νmax 3340 (OH), 3259 (NH), 3065 (CHₐromatic), 2960 cm⁻¹ (CHₐliph); ¹H NMR (CDCl₃) δ (ppm): 1.24 (9H, s, Bu'), 1.29 (27H, s, Bu'), 2.42 (3H, s, CH₃),
5.26 (2H, s, CH₂), 7.32 (2H, d, CH₃on), 7.68 (6H, d, Ar-H), 7.75 (2H, s, CH₃on), 7.95 (2H, d, CH₃on), 9.18 (2H, s, OH), 9.51 (1H, s, OH), 12.22 (1H, s, NH); ¹³C NMR (CDCl₃) δ (ppm): 21.2, 31.0, 31.2, 31.3, 34.2, 34.3, 34.6, 82.5 (exchangeable), 120.0, 120.6, 124.0, 128.3, 129.4, 136.0, 136.4, 136.7, 137.1, 144.5, 144.6, 150.7, 155.8, 156.2, 157.0, 192.9.

A combination of Lawesson's reagent (0.44 gm, 1.11 mmole) and mono-p-(acetylphenyl)acetamide-TCA 3 (1gm, 1.15 mmole) refluxed in 10 mL of benzene while stirring for overnight. A whitish yellow solid product was precipitated, filtered, and recrystallized from methylene chloride and methanol (2:1). (6, C₅₀H₇₅NO₅S₆) Yield 68%; Whitish yellow, mp. 255 °C; MS: m/z 927.26 [MH]⁺; IR (KBr): νmax 3354 (OH) 3332 (NH) cm⁻¹; ¹H NMR (CDCl₃) δ (ppm): 1.23 (9H, s, Bu'), 1.28 (27H, s, Bu'), 2.25 (3H, s, CH₃), 5.24 (2H, s, CH₂), 7.67-7.75 (9H, m, CH₃on), 8.10 (1H, d, CH₃on), 8.32 (1H, d, CH₃on), 9.19 (2H, s, OH), 9.52 (1H, s, OH), 12.37 (1H, s, NH); ¹³C NMR (CDCl₃) δ (ppm): 26.7, 31.0, 31.3, 31.3, 34.2, 34.4, 34.7, 82.7, 119.9, 120.5, 120.6, 123.4, 128.1, 129.3, 135.1, 136.1, 136.6, 137.2, 142.6, 144.6, 144.7, 150.9, 155.7, 156.1, 156.8, 194.0, 197.1.

A mixture of mono-p-(phenacetyl) bromide)acetamide-TCA 4 (1gm, 1.02 mmole) and Lawesson’s reagent (0.41 gm, 1.03 mmole) in 10 mL benzene was stirred under reflux overnight. A whitish-yellow solid product was precipitated, filtered, and recrystallized from methylene chloride and methanol (2:1). (7, C₅₀H₇₅BrNO₅S₆) Yield 58%; Whitish yellow, mp 190 °C; MS: m/z 1007.26 [MH]⁺; IR (KBr): νmax 3473 (OH), 3360 (NH), 3062 (CH₃on), 2960 (CH₃) cm⁻¹; ¹H NMR (CDCl₃) δ (ppm): 1.12 (9H, s, Bu'), 1.18 (27H, s, Bu'), 3.78 (2H, s, CH₂), 5.13 (2H, s, CH₂), 7.56-7.64 (10H, m, CH₃on), 8.09-8.11 (2H, m, CH₃on), 9.09 (2H, s, OH), 9.52 (1H, s, OH), 12.19 (1H, s, NH).

2.2. Insect Collection and Rearing

The cowpea aphid bug is a very dangerous and destructive pest that attacks a wide variety of Egypt’s agricultural crops. The initial batches of Aphis craccivora insects were taken from the Agricultural Research Center, Plant Protection Research Institute, and Pest Laboratory. A. craccivora lab strain was used to test the newly generated compounds' insecticidal efficacy.

2.3. Laboratory Bioassay.

Under the same laboratory conditions, all six derivatives were examined using the leaf dipping method to demonstrate their insecticidal activities [20]. In the control experiment, only water, acetone, and 0.1% Tween-80 were used. Different concentrations of each derivative were utilized (in acetone and 0.1% Tween-80 as a surfactant was used). Twenty nymphs and twenty adults of A. craccivora, three times, roughly the same size, were submerged for 10 seconds in each analogous concentration. The test aphids were left to dry at room temperature for about half an hour. These toxicological experiments were conducted at 25 °C with 5% relative humidity. The used pests were transferred to glass jars filled with water once they had dried. Using a modern binocular microscope, the aphid mortality was determined following a single day of therapy. Because it was immobile, the aphid was considered dead. Using Abbott's formula, all of the synthetic derivatives' mortality data were examined. [21]. Insecticidal activity evaluation is dependent on LC50 values, which are determined by probit analysis [22].

5. Conclusion

Synthesis of a novel compound of monosubstituted thiaaxarene derivatives used as insecticidal chemicals against Cowpea Aphid adults and nymphs.

CRediT authorship contribution statement:

Data availability statement
The data used to support the findings of this study are available from the corresponding author upon request.

Declaration of competing interest
The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

References

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Sohag J. Sci. 2024, 9(4), 423-428 427