New Dibenzothiepine Sulfones
Synthesis and structure elucidation

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Some new dibenzothiepine sulfones were obtained by acylation of 2-methyl-11-hydroxymino-6,11-dihydrodibenzo[b,e]thiepin-5,5-dioxide with various acid chlorides. The synthesis of the intermediate oxime was performed in several stages. Thus, by reaction of phthalide with potassium p-thiocresolate, was obtained 2-(4-tolyliothio)methyl)benzonic acid. The acid was cyclized to the desired 2-methyl-6,11-dihydrodibenzo[b,e]thiepin-11(6H)-one in the presence of polyphosphoric acid, converted afterwards to the corresponding 5,5-dioxide and subsequently to the corresponding oxime. All the new compounds have been characterized by elemental analysis, spectral analysis (¹H-NMR, ¹³C-NMR, IR) and thin layer chromatography (TLC).

Keywords: sulfones, dibenzo[b,e]thiepine, NMR spectroscopy

The interest for the tricyclic compounds having dibenzothiepine structure has been heightened during the last years due to their broad spectrum of biological activities. Dibenzothiepine derivatives have significant psychotropic activities [1, 2], but also other important properties. Zaltoprofen is a nonsteroidal anti-inflammatory drug with powerful analgesic action [3]. Methiotepine and octochlorethine, two dibenzothiepine antipsychotics, were found to have significant bacteriostatic activity towards M. tuberculosis, including virulent strains [4]. Some dibenzothiepine compounds demonstrated their potential use as antiprotozoal agents [5] and more recently dibenzo[b,e]thiepines proved to be active as inhibitors of tumor necrosis factor-α (TNF-α), and therefore useful for the treatment and prevention of disorders caused by increased TNF-α activity, in particular inflammations [6]. Researchers have also reported [7] that some dibenzothiepine derivatives are useful as insecticidal, acaricidal and nematicidal agents.

Motivated by these findings and as part of our ongoing studies, we proposed to synthesize new biologically active dibenzo[b,e]thiepine derivatives. In previous papers we reported the synthesis and antipathogenic activity of some dibenzothiepine, against planktonic cells and bacterial cells grown in biofilms [8-12]. We also reported the results of in silico evaluations performed on dibenzothiepine compounds [13]. The predictions were focused on molecular descriptors relevant for quantitative estimation of penetration across various biological barriers. The results indicated a low solubility - high permeability profile, with considerable impact of gastro-intestinal, endogenous and intestinal characteristics proved to be favorable for expressing a potential psychotropic activity for dibenzothiepine compounds.

Experimental part
All chemicals used were purchased from commercial sources (Merck, Fluka, Sigma - Aldrich) and were of reagent grade. Solvents were used as received, except benzene (dried over sodium and then distilled) and pyridine (stored over potassium hydroxide and then distilled).

Melting points were determined with an Electrothermal 9100 apparatus in open capillary tubes and are uncorrected. Elemental analyses were carried out using a Perkin Elmer CHNS/O Analyzer Series II 2400 apparatus, and the results were within ±0.4% of theoretical values.

Thin layer chromatography (TLC) was performed on silicagel 60F254 Merck plates. For the development chloroform/ethyl acetate (10:1) was used. The visualization was performed using an UV lamp (λ = 254 nm) and iodine atmosphere.

The IR spectra were recorded using a FT-IR Bruker Vertex 70 spectrometer, with horizontal device for attenuated reflectance and diamond crystal, on a spectral ranging from 4000 to 400 cm⁻¹, at a spectral resolution of 2 cm⁻¹. The IR bands are denoted as: w-weak; m- medium; s- strong; vs- very strong.

The NMR spectra were recorded on a Varian INOVA 400 spectrometer operating at 9.4 Tesla, corresponding to the resonance frequency of 399.95 MHz for the ¹H nucleus and 100.56 MHz for the ¹³C nucleus. TMS was used as internal standard both in proton and carbon spectra. All the spectra were recorded at 303K with an indirect detection probehead AS-SW and field gradients.

The ¹H-NMR data are reported in the following order: chemical shift (ppm), multiplicity number of protons, assignment of the signal, coupling constant (J) in hertz. The splitting patterns are abbreviated as following: s, singlet; bs, broad singlet; d, doublet; bd, broad doublet; dd, double doublet; ddd, doublet of double doublets; dq, double quartet; t, triplet; td, triple doublet.

The ¹³C-NMR data are reported in the following order: chemical shift (ppm), the signal / atom attribution, the coupling constant (J) in some cases; (Cq- quaternary carbon).

The precursors, 2-(4-tolyliothio)methyl)benzonic acid (3), 2-methyl-6,11-dihydrodibenzo[b,e]thiepin-11(6H)-one (4)
2-methyl-6,11-dihydropyridobenzo\(b,e\)thiepin-11-one 5,5-dioxide (5) and 2-methyl-11-hydroxymino-6,11-dihydropyridobenzo\(b,e\)thiepin-5,5-dioxide (6) were synthesized according to previously described procedures [11, 12, 14, 15].

**General procedure for synthesis of 2-methyl-O-acyl-oximino-dibenzo\[b,e\]thiepin-5,5-dioxides (7a-i)**

A solution of 0.01 mol of acetylated acetic chloride in 10 mL anhydrous benzene together with 0.8 mL (0.79 g; 0.01 mol) dry pyridine (Mol wt 79.098; d²=0.978) was added dropwise to a solution 2-methyl-11-hydroxymino-6,11-dihydropyridobenzo\(b,e\)thiepin-5,5-dioxide (M, 287.34) (0.01 mol) in anhydrous benzene. The reaction mixture was refluxed for two hours, cooled, the precipitate was filtered off and the solvent removed under reduced pressure. The resulting crude product was recrystallized from an appropriate solvent (ethanol/glacial acetic acid).

Spectral data for the new compounds (7a-i).

\{\[5,5\text{-dioxo-dibenzo\[b,e\]thiepin-2-methyl-11(6H)-yliden][amino][oxy]ethyl\}methanone (7a)

From the spectra results that only one stereoisomer is present.

**FT-IR** (ATR in solid, ν cm⁻¹): 3061w; 2960w; 2920w; 1765vs; 1590m; 1472m; 1436m; 1411w; 1306v; 1267s; 1254s; 1238vs; 1196w; 1154s; 1123s; 1090s; 1064w; 1037s; 1095s; 987m; 916m; 863m; 823m; 783m; 746m; 713m; 649m; 485m.

**¹H-NMR** (CDCl₃, δ ppm, J Hz): 7.92 (d, 1H, H-4, 8.0); 7.70 (d, 1H, H-1, 1.0); 5.78 (dd, 1H, H-10, 1.6, 7.4); 7.53 ÷ 7.36 (m, 7H, aromatic protons); 7.25 (m, 1H, H-16); 5.09 (bs, H-6A); 4.34 (bs, H-6B); 2.47 (s, H-2').

**¹³C-NMR** (CDCl₃, δ ppm, J Hz): 165.16(C-11); 162.70(C-12); 143.73(Cq); 138.98(Cq); 134.98(Cq); 135.99(Cq); 129.65(Cq); 128.11(Cq); 124.17(Cq); 133.26(CH); 132.95(CH); 131.59(C-10); 131.31(CH); 131.20(CH); 130.90(CH); 129.99(C-1); 129.03(CH); 127.80(CH); 126.71(C-17); 126.23(C-4); 58.35(C-6); 21.36(C-2').

\{\[1\:(E,Z)-(5,5\text{-dioxo-dibenzo\[b,e\]thiepin-2-methyl-11(6H)-yliden)[amino][oxy]-(3-chloro-phenyl)methanone (7d)

Syn-anti isomers mixture in ratio 1:2.2.

**FT-IR** (ATR in solid, ν cm⁻¹): 3069w; 2972w; 2924w; 1763vs; 1303vs; 1156s; 1233vs; 1127s; 1080m; 1056s; 987m; 873m; 781w; 768w; 739m; 515w.

**¹H-NMR** (CDCl₃, δ ppm, J Hz): 8.20(d, 1H, H-4, 8.2); 7.94(d, 1H, H-4, 8.2); 7.92(d, 1H, H-4, 8.0); 7.82 ÷ 7.30 (m, 7H, H- arom); 7.70(d, 1H, H-1, 1.0); 7.58(dd, 1H, H-10, 1.6, 7.4); 7.53 ÷ 7.36 (m, 7H, H- arom); 7.25 (m, 1H, H-16); 5.07 (bs, H-6A); 4.35(bs, H-6B); 2.53(s, H-2').

**¹³C-NMR** (CDCl₃, δ ppm, J Hz): 164.94(C-11); 161.96(C-12); 143.78(Cq); 139.00(Cq); 134.84(Cq); 134.68(Cq); 133.79(CH); 133.03(CH); 131.60(CH); 131.09(CH); 130.14(CH); 129.99(CH); 129.82(CH); 129.56(Cq); 129.94(CH); 127.76(CH); 127.69(CH); 126.66(CH); 126.27(CH); 124.37(CH); 59.25(C-6); 58.54(C-6); 21.38(C-2').

\{\[5,5\text{-dioxo-dibenzo\[b,e\]thiepin-2-methyl-11(6H)-yliden)[amino][oxy]-(4-chloro-phenyl)methanone (7e)

From the spectra results that only one stereoisomer is present.

**FT-IR** (ATR in solid, ν cm⁻¹): 3053w; 2969w; 2923w; 1747vs; 1591s; 1483m; 1451w; 1400m; 1304vs; 1242v; 1194m; 1155s; 1124m; 1089m; 1075s; 1049s; 992s; 914m; 877w; 786m; 779w; 746m; 694m; 680m; 657w; 630w; 552w; 529w; 514m.

**¹H-NMR** (CDCl₃, δ ppm, J Hz): 7.93(d, 1H, H-4, 8.3); 7.69(d, 1H, H-1, 1.4); 7.68(d, 2H, H-14, H-18, 8.9); 7.54(dd, 1H, H-1, 1.4, 8.3); 7.53 ÷ 7.47 (m, aromatic protons); 7.40(m, 1H, H-7); 7.35(d, 2H, H-15, H-17, 8.9); 5.06(bs, 1H, H-6A); 4.35(bs, 1H, H-6B); 2.48(s, H-2').

**¹³C-NMR** (CDCl₃, δ ppm, J Hz): 164.72(C-11); 162.35(C-12); 143.77(Cq); 140.41(Cq); 139.05(Cq); 134.83(Cq); 132.99(CH); 131.59(C-3); 131.06(C-14, C-18); 131.02(C-1); 130.15(CH); 129.64(CH); 129.09(C-15, C-17); 128.92(CH); 127.67(C-7); 126.42(CH); 126.28(C-4); 124.47(C-1a); 58.58(C-6); 21.38(C-2').

\{\[1\:(E,Z)-(5,5\text{-dioxo-dibenzo\[b,e\]thiepin-2-methyl-11(6H)-yliden)[amino][oxy]-(3-bromo-phenyl)methanone (7f)

Syn-anti isomers mixture in ratio 1:3.3.

**FT-IR** (ATR in solid, ν cm⁻¹): 3371w; 3065w; 2968w; 2923w; 1761vs; 1590w; 1567w; 1450w; 1423w; 1301s; 1259m; 1231vs; 1197m; 1154s; 1123s; 1073s; 1053m; 1019w; 994w; 918m; 867m; 820m; 780m; 739s; 694v; 515m.

**¹H-NMR** (CDCl₃, δ ppm, J Hz): 7.94(d, 1H, H-2, 8.2); 7.86(d, 1H, H-14, 1.8); 7.80(m, 1H, H-16); 7.69(m, 2H, aromatic protons).
protons); 7.58 + 7.30 (m, 5H, aromatic protons); 7.26 (t, 1H, H-17, 7.9); 5.07 (s, H-6A); 4.35 (s, H-6B); 2.53 (s, H-2'M); 2.46 (s, H-2'M).

13C-NMR (CDCl3, δ ppm): 164.98 (C-11); 161.83 (C-12); 143.79 (Cq); 139.02 (Cq); 136.70 (CH); 134.68 (Cq); 133.03 (CH); 132.77 (CH); 131.61 (CH); 130.26 (CH); 129.87 (Cq); 129.56 (Cq); 128.95 (CH); 128.22 (CH); 127.71 (CH); 126.29 (CH); 124.38 (Cq); 122.71 (CH); 59.26 (C-6); 58.55 (C-6'); 21.56 (C-2''M); 21.38 (C-2'M).

\{[5,5-dioxo-dibenzo[b,e]thiepin-2-methyl-11(6H)-ylidenaminoxy](2,3-dimethoxy-phenyl)methanone (7g)

Syn-anti isomers mixture in ratio 1:3.8.

FT-IR (ATR in solid, ν cm⁻¹): 3061w; 2968w; 2927w; 1755vs; 1738vs; 1579w; 1561w; 1326m; 1305vs; 1262m; 1247w; 1238m; 1228m; 1217w; 1150s; 1045m; 780w; 770s; 743m.

1H-NMR (CDCl3, δ ppm, J Hz): 7.90 (d, 1H, H-4, 8.2); 7.61 (bd, 1H, H-1, 1.0); 7.29 (d, 1H, H-16, 8.4); 7.28 + 7.50 (m, 5H, H-arom); 6.59 (d, 1H, H-15, 8.4); 6.51 (d, 1H, H-17, 8.4); 5.20 (bs, 1H, sis AB, H-6B); 4.36 (bs, 1H, sis AB, H-6A); 3.75 (s, 6H, -OCH3); 2.46 (s, 3H, H-2'M).

13C-NMR (CDCl3, δ ppm): 165.34 (C-11); 163.48 (C-12); 143.74 (Cq); 141.53 (C-15'); 141.36 (C-15''); 139.06 (Cq); 134.93 (Cq); 133.39 (CH m); 133.18 (CH M); 132.99 (CH); 131.84 (Cq); 131.45 (CH); 131.09 (CH); 131.02 (CH); 130.98 (CH); 130.91 (CH); 130.08 (CH); 129.69 (Cq); 129.09 (CH); 127.94 (CH); 127.76 (CH); 126.50 (CH''); 126.28 (CH''); 124.43 (Cq); 94.20 (C-16); 59.26 (C-6); 58.55 (C-6'); 21.56 (C-2''M); 21.38 (C-2'M).

\{[5,5-dioxo-dibenzo[b,e]thiepin-2-methyl-11(6H)-ylidenaminoxy](2-iodo-phenyl)methanone (7h)

From the spectra results that only one stereoisomer is present.

FT-IR (ATR in solid, ν cm⁻¹): 3017w; 2978w; 2944w; 2837w; 1763vs; 1594s; 1430m; 1169s; 1035s; 743m.

1H-NMR (CDCl3, δ ppm, J Hz): 7.90 (d, 1H, H-4, 8.2); 7.61 (bd, 1H, H-1, 1.0); 7.29 (d, 1H, H-16, 8.4); 7.28 + 7.50 (m, 5H, H-arom); 6.59 (d, 1H, H-15, 8.4); 6.51 (d, 1H, H-17, 8.4); 5.20 (bs, 1H, sis AB, H-6B); 4.36 (bs, 1H, sis AB, H-6A); 3.75 (s, 6H, -OCH3); 2.46 (s, 3H, H-2'M).

13C-NMR (CDCl3, δ ppm): 164.35 (C-12); 162.15 (C-11); 157.97 (Cq); 157.87 (Cq); 153.57 (Cq); 130.04 (Cq); 132.03 (CH); 131.07 (CH); 130.55 (CH); 130.23 (CH); 129.98 (Cq); 129.08 (Cq); 128.66 (CH); 128.65 (CH); 128.14 (CH); 126.05 (CH); 124.19 (Cq); 124.19 (Cq); 103.78 (CH); 103.78 (CH); 58.40 (C-6); 55.88 (-OCH3); 21.33 (C-2''M).

Results and discussions

The synthetic route for the newly synthesized sulfones is illustrated in scheme 1.

The key intermediate, 2-methyl-11-hydroxyimino-6,11-dihyrodibenzo[b,e]thiepin (6), was prepared, in several stages, according to the previously described procedure [12, 15]. Thus, by reaction of phthalide (1) with potassium thiosulfitic acid in xylene under reflux, was obtained the acid (3). The resulted potassium salt of 2-(4-tolyliothio)methylbenzoic acid showed a good solubility in an aqueous solution of 10% potassium hydroxide and was separated from xylene through precipitation upon acidification using a mineral acid solution. Potassium p-thiocrresolate (2), in xylene under reflux, was obtained the acid (3). The resulted potassium salt of 2-(4-tolyliothio)methylbenzoic acid showed a good solubility in an aqueous solution of 10% potassium hydroxide and was separated from xylene through precipitation upon acidification using a mineral acid solution.

The synthetic route for the newly synthesized sulfones (7a-i) was prepared by cyclodehydration of the corresponding oxime (3) in the presence of polyphosphoric acid (PPA). The aforementioned ketone (4) was synthesized by cyclodehydration of acid (3) in the presence of polyphosphoric acid (PPA).

2-Methyl-6,11-dihyrdobienzo[b,e]thiepin-11(6H)-one (4) was synthesized by cyclodehydration of acid (3) in the presence of polyphosphoric acid (PPA).

The new dibenzothiepine sulfones (7a-i) were prepared by acylation of the 2-methyl-11-hydroxyimino-6,11-dihydrdibenzo[b,e]thiepin-5,5-dioxide (6) with various acid chlorides, in dry benzene or toluene and in the presence of anhydrous pyridine as proton acceptor.

Scheme 1. Synthesis of the new dibenzothiepine sulfones

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It should be noted that our attempt to obtain \((\text{11(E,Z)-5,5-dioxo-dibenzo}[b,e]\text{thiepin-2-methyl-11(6H)-yliden}][\text{amino}][\text{oxy}]\text{ethylmethanone (7a, } R = -\text{CH}_2\text{-CH}_3\) following the synthesis pathway presented in our previous communication \[11\] (Scheme 2), via oxime (8) and O-acyloximino-dibenzo[b,e]thiepin (9, \(R = -\text{CH}_2\text{-CH}_3\)), was not successful. In the last stage, under the reaction condition dibenzo[b,e]thiepin-2-methyl-11(6H)-yliden][amino][oxy] ethylmethanone (9, \(R = -\text{CH}_2\text{-CH}_3\)) was cleaved, affording the oxidation product, oxime 6.

The structure, some physical properties and the elemental analysis of the new dibenzothiepine sulfones 7a-i are presented in Table I. All elemental analyses results were within ±0.4% of the theoretical values.

The new dibenzothiepine sulfones are solid, crystallized, white or light yellow, soluble at room temperature in acetone, chloroform, benzene, toluene, xylene, dichloromethane, by heating in inferior alcohols, insoluble in water.

In thin layer chromatography (TLC) we found single spot, some time with tailing, with different Rf values for each compound. The single spot indicates that synthesized compounds are pure and contain little or no impurities.

The structures of the newly synthesized compounds were elucidated by spectral data. The IR, \(^1\text{H}-\text{NMR}\) and \(^{13}\text{C}-\text{NMR}\) spectra show all the expected signals.

The presence of the sulphur atom induces the asymmetry in dibenzothiepine nucleus, so the oxime (6) may have \text{syn} or \text{anti} configuration. Therefore, a notable difference appears in some compounds (7a-i) between the chemical shifting of the protons from the dibenzo rings. Also, the number of the carbon signals is larger than that corresponding to the raw formula, owing to the two different \text{sin} and \text{anti} configurations. As to the carbon atoms data, the chemical shifting have been noted by \(M\) for the major and \(m\) from the minor compound.

### Table I

CHARACTERIZATION DATA OF COMPOUNDS 7a-i

<table>
<thead>
<tr>
<th>Compd</th>
<th>(R)</th>
<th>Molecular formula</th>
<th>Molecular mass</th>
<th>M.p. (°C)</th>
<th>Yield (%)</th>
<th>Rf</th>
<th>Elemental analysis (calc/found)</th>
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<tr>
<td>7a</td>
<td>-\text{CH}_2\text{-CH}_3</td>
<td>(\text{C}<em>{18}\text{H}</em>{17}\text{NO}_3\text{S})</td>
<td>343.40</td>
<td>164.1-165.9</td>
<td>79.3</td>
<td>0.64</td>
<td>62.96/62.73, 4.99/4.78, 4.08/4.35, 9.34/9.67</td>
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<tr>
<td>7b</td>
<td>2-F-\text{C}_6\text{H}_4</td>
<td>(\text{C}<em>{22}\text{H}</em>{18}\text{FNO}_3\text{S})</td>
<td>409.43</td>
<td>190.1-194.2</td>
<td>78.2</td>
<td>0.68</td>
<td>64.54/63.89, 3.94/3.88, 3.42/3.45, 7.83/7.85</td>
</tr>
<tr>
<td>7c</td>
<td>2-Cl-\text{C}_6\text{H}_4</td>
<td>(\text{C}<em>{22}\text{H}</em>{16}\text{ClNO}_3\text{S})</td>
<td>425.88</td>
<td>220.0-227.0</td>
<td>81.5</td>
<td>0.65</td>
<td>62.05/61.74, 3.79/3.65, 3.29/3.32, 7.53/7.62</td>
</tr>
<tr>
<td>7d</td>
<td>3-Cl-\text{C}_6\text{H}_4</td>
<td>(\text{C}<em>{22}\text{H}</em>{16}\text{ClNO}_3\text{S})</td>
<td>425.88</td>
<td>186.3-190.1</td>
<td>80.6</td>
<td>0.67</td>
<td>62.05/61.69, 3.79/3.68, 3.29/3.31, 7.53/7.58</td>
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<td>7e</td>
<td>4-Cl-\text{C}_6\text{H}_4</td>
<td>(\text{C}<em>{22}\text{H}</em>{16}\text{ClNO}_3\text{S})</td>
<td>425.88</td>
<td>229.3-236.2</td>
<td>84.6</td>
<td>0.74</td>
<td>62.05/62.03, 3.79/3.84, 3.29/3.32, 7.53/7.48</td>
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<tr>
<td>7f</td>
<td>3-Br-\text{C}_6\text{H}_4</td>
<td>(\text{C}<em>{22}\text{H}</em>{16}\text{BrNO}_3\text{S})</td>
<td>470.33</td>
<td>160.3-164.2</td>
<td>79.4</td>
<td>0.69</td>
<td>56.18/56.30, 3.43/3.48, 2.98/3.00, 6.82/6.84</td>
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<td>7g</td>
<td>2-I-\text{C}_6\text{H}_4</td>
<td>(\text{C}<em>{22}\text{H}</em>{16}\text{INO}_3\text{S})</td>
<td>517.33</td>
<td>186.4-192.0</td>
<td>71.3</td>
<td>0.78</td>
<td>51.08/50.60, 3.12/3.20, 2.71/2.72, 6.20/6.22</td>
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<tr>
<td>7h</td>
<td>2,3-(\text{OCH}_3)_2-\text{C}_6\text{H}_3</td>
<td>(\text{C}<em>{24}\text{H}</em>{21}\text{NO}_3\text{S})</td>
<td>451.50</td>
<td>196.8-198.7</td>
<td>86.6</td>
<td>0.71</td>
<td>63.85/63.58, 4.69/4.91, 3.10/3.42, 7.10/7.38</td>
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<tr>
<td>7i</td>
<td>2,6-(\text{OCH}_3)_2-\text{C}_6\text{H}_3</td>
<td>(\text{C}<em>{24}\text{H}</em>{21}\text{NO}_3\text{S})</td>
<td>451.50</td>
<td>237.8-239.9</td>
<td>64.4</td>
<td>0.72</td>
<td>63.85/63.96, 4.69/4.52, 3.10/3.39, 7.10/7.42</td>
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Conclusions

In this study we report the synthesis and characterization of new compounds from dibenzothiepine class. The target compounds were obtained by acylation of 2-methyl-11-hydroxyimino-6,11-dihydrodibenzo[b,e]thiepin-5,5-dioxide with various acid chlorides. The synthesis of the intermediate oxime was performed in several stages. Thus, by reaction of phtalide with thiophenol potassium salt, was obtained 2-(phenylthiomethyl)benzoic acid. The acid was cyclized to the desired 6,11-dihydrodibenzo[b,e]thiepin-11(6H)-one in the presence of polyphosphoric acid, converted afterwards to the corresponding 5,5-dioxide and subsequently to the corresponding oxime.

For the prepared set of compounds were performed melting point determination, Rf value determination and solubility profile. The structures elucidation of the newly dibenzothiepine sulfones was carried out by different spectroscopic techniques 1H-NMR, 13C-NMR, IR. Further confirmations of the compounds were carried out by elemental analysis.

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