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Synthesis and Stereochemistry of Some New 1,3-Oxathiane Derivatives

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Summary. The synthesis and stereochemistry of some new 2,5-substituted 1,3-oxathiane derivatives are reported. The anancomeric or flexible structure of the derivatives and some peculiar cases of prochirality are revealed by NMR investigations.

Keywords. Heterocycles; Conformation; Prochirality; NMR spectroscopy; Diastereotopicity.

Introduction

The stereochemistry of 1,3-oxathiane derivatives [1–5] is less studied than the stereochemistry of 1,3-dioxanes [6–8] or 1,3-dithianes [2], [9], mainly due to the relatively difficult access to 3-mercapto-1-propanol synthones and to the complex stereochemistry of the heterocycles bearing different heteroatoms in the ring.

The reported investigations on the stereochemistry of 1,3-oxathiane derivatives revealed the chirality of the 1,3-oxathiane ring [1] due to the presence of a tricoordinated virtual chiral center (Scheme 1, [10]).

The stereochemistry investigations of 1,3-oxathiane derivatives pointed out that the majority of the properties of these compounds exhibit values close to the average of the values of the same properties measured in similar 1,3-dioxane and 1,3-dithiane derivatives [1]. The rather small number of papers on the stereochemistry of the 1,3-oxathiane ring and the complex configuration and conformation behavior of this heterocycle justify the interest for investigations on the structural aspects of some new series of derivatives.

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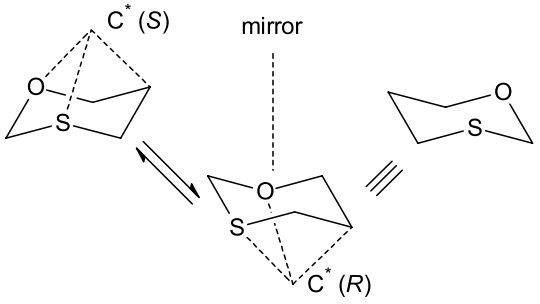
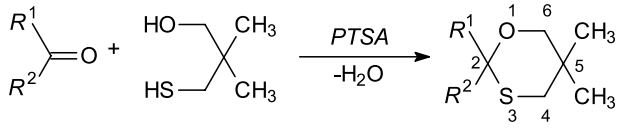


Scheme 1

Results and Discussions

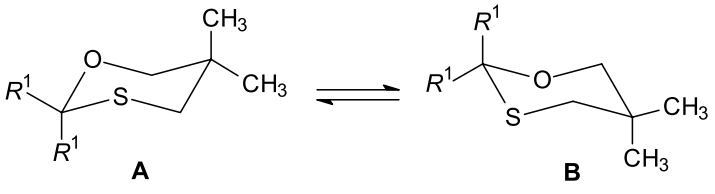
2,5-Substituted 1,3-oxathiane derivatives **2**–**12** were obtained by condensation of mercaptopropanol (**1**) with several aldehydes and ketones (Scheme 2).

Compounds **2** and **3** exhibit flexible structures, due to a fast flipping of the 1,3-oxathiane ring (Scheme 3). The NMR spectra (Table 1 and Experimental) exhibit unique signals for the axial and equatorial orientations of the protons of the hetero-



1	$R^1 = R^2 = C_2H_5$	**2**
	$R^1 = R^2 = CH_2\text{-}C_6H_5$	**3**
	$R^1 = H$; $R^2 = i\text{-}C_3H_7$	**4**
	$R^1 = H$; $R^2 = C_6H_5$	**5**
	$R^1 = H$; $R^2 = 1\text{-}C_{10}H_7$	**6**
	$R^1 = H$; $R^2 = 2\text{-}C_{10}H_7$	**7**
	$R^1 = CH_3$; $R^2 = C_6H_5$	**8**
	$R^1 = CH_3$; $R^2 = p\text{-}C_6H_4\text{-}NO_2$	**9**
	$R^1 = CH_3$; $R^2 = 1\text{-}C_{10}H_7$	**10**
	$R^1 = CH_3$; $R^2 = 2\text{-}C_{10}H_7$	**11**
	$R^1 = C_2H_5$; $R^2 = C_6H_5$	**12**

Scheme 2



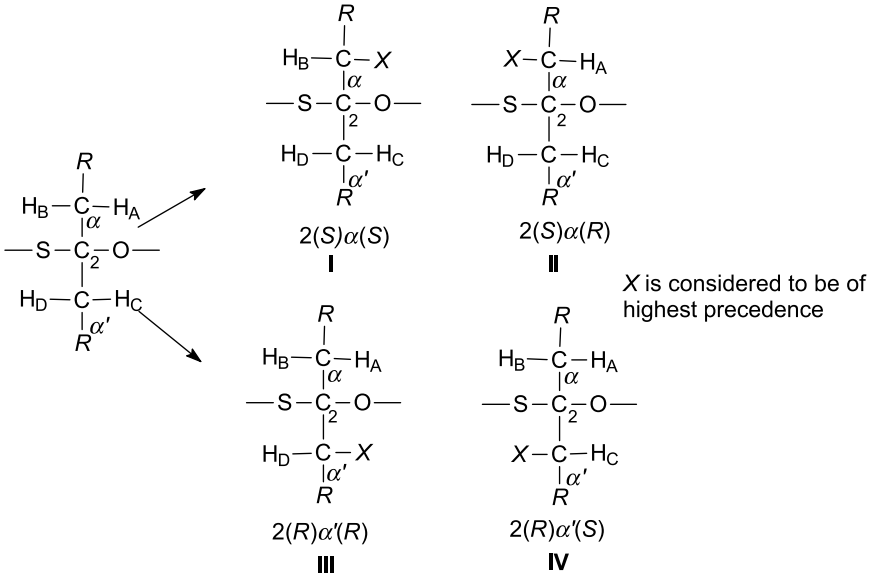
Scheme 3

Table 1. ^{1}H NMR ($CDCl_3$, δ, ppm) data for compounds **2**–**12**

Compound	4-H		6-H		5-CH_3	
	eq.	ax.	eq.	ax.	eq.	ax.
2	2.58		3.37		1.03	
3	2.44		3.62		0.93	
4	2.39	2.84	3.59	3.25	0.84	1.20
5	2.47	3.20	3.78	3.49	0.97	1.40
6	2.56	3.24	3.89	3.65	1.00	1.44
7	2.52	3.15	3.83	3.55	0.97	1.40
8	2.31	2.60	3.36	3.29	0.78	1.28
9	2.36	2.52	3.42	3.21	0.81	1.26
10	2.33	2.88	3.31	3.06	0.67	1.36
11	2.33	2.68	3.39	3.31	0.75	1.31
12	2.32	2.57	3.32	3.32	0.77	1.27

cycle or of the similar groups located on it. The flipping of the 1,3-oxathiane ring is an enantiomeric inversion.

An interesting feature in the NMR spectra of **2** and **3** are different signals for the methylene protons of the $-CH_2-R$ ($R = CH_3$, C_6H_5) groups at position 2. These protons exhibit different signals despite the flipping of the 1,3-oxathiane ring and despite missing a conformationally constant chiral element. Careful inspection of structures **I**–**IV** obtained using the substitution test (Scheme 4) shows that the replacement of a hydrogen atom at the prochiral centers α and α' determines besides the transformation of these centers in chiral carbon atoms, the simultaneous transformation of the carbon atom at position 2 in another chiral center.



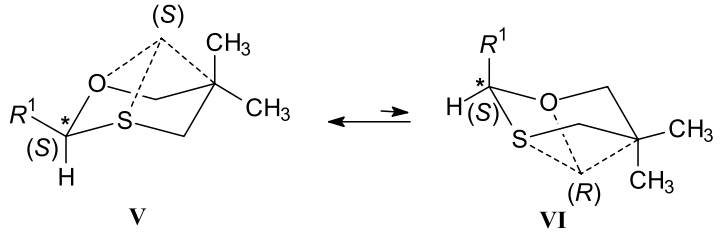
Scheme 4

Structures **I**–**IV** exhibit two chiral elements and they form two pairs of enantiomers representing two diastereoisomers (*like*: **I**, **III** and *unlike*: **II**, **IV**). In conclusion protons A and C (B and D) are enantiotopic and A or C and B or D are diastereotopic.

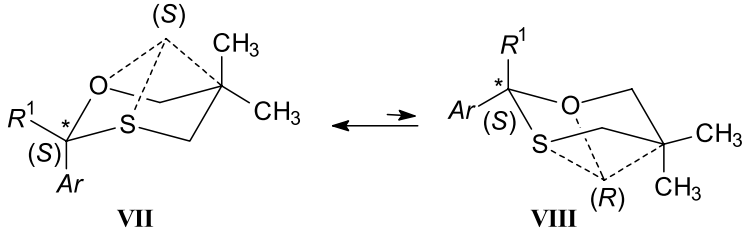
Due to the flexibility of the heterocycle the ^{1}H NMR spectrum of **3** exhibits a singlet for the methyl groups at position 5 ($\delta_{Me} = 0.93$ ppm) and splitted singlets (doublets, due to the long range coupling with the protons of the benzyl groups; $^5J = 1.7$ Hz) for the protons at positions 4 ($\delta_4 = 2.44$ ppm) and 6 ($\delta_6 = 3.62$ ppm). The methylene protons of the benzyl groups exhibit two well separated doublets of doublets ($\delta_{A,C} = 3.03$; $\delta_{B,D} = 3.25$ ppm) with large geminal ($J = 14.3$ Hz) and small long range ($^5J = 1.7$ Hz) coupling constants. The diastereotopicity ($\Delta\delta_{A,C-B,D}$) for the 2-CH_2-protons for compound **3** ($\Delta\delta$(**3**) = 0.22 ppm) is somewhat larger than the same difference for **2** ($\Delta\delta$(**2**) = 0.13 ppm).

Compounds **4**–**12** exhibit anancomeric structures. The conformational equilibria for compounds **4**–**7** are shifted towards the conformer exhibiting the substituent at position 2 in equatorial orientation (*V*, Scheme 5) in agreement with the high A values for alkyl and aryl groups at position 2 [11, 12] and with the results of R*O*ESY spectra (run with **4** and **5**) that show important interactions between the signals belonging to the proton at position 2 (axial orientation) and the signals of the axial protons of the heterocycle (positions 4 and 6).

The conformational equilibria in **8**–**12** are shifted towards the conformer bearing the aromatic group in axial orientation (Scheme 6). As in similar 2-alkyl,2-aryl-1,3-dioxane derivatives the aromatic group in compounds **8**–**12** strongly prefers the axial orientation. The R*O*ESY spectra of **9** and **10** revealed weak interactions between the axial protons of the heterocycle and the methyl group at position 2 and important interactions of the same protons with the protons of the aromatic



Scheme 5



Scheme 6

group at position 2 proving the axial orientation of the aromatic substituents at position 2.

The NMR spectra of the compounds (Table 1) exhibit different signals for the axial and equatorial protons of the heterocycle and for the axial and equatorial methyl groups at position 5. As an example, the spectrum of **5** exhibits more deshielded signals for the axial groups or protons at positions 5 and 4 ($\delta_{5Me(ax)} = 1.40$; $\delta_{5Me(eq)} = 0.97$, $\delta_{4ax} = 3.20$; $\delta_{4eq} = 2.47$ ppm) and for the equatorial ones at position 6 ($\delta_{6ax} = 3.49$; $\delta_{6eq} = 3.78$ ppm). The axial aromatic group prefers the orthogonal orientation and the magnetic anisotropy of the aromatic ring determines a shielding of the protons of the methyl group located at position 5 and of the protons of the heterocycle.

The carbon atom at position 2 is chiral but of course **4–12** are obtained as racemic mixtures. The conformational preference of the substituent at position 2 and the configuration of this chiral carbon atom determine the preferred configuration of the heterocycle (Schemes 5 and 6).

Conclusions

The synthesis and the stereochemistry of 2,5-substituted 1,3-oxathiane derivatives is reported. The NMR investigations reveal flexible and anancomeric structures. The aromatic group in 2-aryl-1,3-oxathianes prefers the equatorial orientation, while in 2-aryl,2-alkyl-derivatives the aromatic group exhibits an axial orientation. The protons of the prochiral center of the substituents located at the position 2 in flipping compounds are diastereotopic and show different signals in NMR spectra.

Experimental

^{1}H and ^{13}C NMR spectra were recorded at room temperature using $CDCl_3$ as solvent in 5 mm tubes on a Varian Gemini spectrometer equipped with a multinuclear head operating at 300 MHz for protons and 75 MHz for carbon atoms. The IR spectra were recorded on a JASCO 630 FTIR spectrometer. The EI mass spectra have been recorded at 70 eV with a GC-MSD System, Hewlett-Packard 5890 II/5972 spectrometer. Melting points were measured with a *Kleinfeld* melting point apparatus and are uncorrected. Elemental analyses (C, H, N, S) were conducted at "Babes-Bolyai" University using a Perkin Elmer Analyser; their results were found to be in good agreement (±0.2%) with the calculated values.

*General Procedure for the Synthesis of Compounds **2–14***

Stoichiometric amounts of 11 mmol of 3-mercaptopropan-1-ol, 11 mmol of carbonyl compound, and catalytic amounts (0.05 g) of *p*-toluenesulfonic acid were refluxed in 20 cm^3 of benzene and H_2O was removed using a *Dean-Stark* trap. After H_2O was separated and the reaction mixture was cooled to room temperature, the catalyst was neutralized under stirring with excess of 0.1 *M* KOH solution. The organic layer was washed twice with 20 cm^3 of H_2O. After drying (Na_2SO_4) the solvent was removed and the oxathiane was purified by vacuum distillation and/or by flash-chromatography.

2,2-Diethyl-5,5-dimethyl-1,3-oxathiane (**2**, $C_{10}H_{20}OS$)

Yield: 45%; colorless liquid; bp 80°C/2 mm Hg; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.91$ (t, $J = 7.2$ Hz, 6H, 2-CH_2-$\underline{CH_3}$), 1.03 (s, 6H, 5-CH_3), 1.80 (dq, $J = 13.5$, 7.2 Hz, 2H, 2-C($\underline{H}$)H′-CH_3), 1.93 (dq,

$J = 13.5$, 7.2 Hz, 2H, 2-C(H)H′-CH_3), 2.58 (s, 2H, H-4), 3.37 (s, 2H, H-6) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 8.07$ (2-CH_2-CH_3), 24.88 (5-CH_3), 27.61 (C-5), 29.16 (2-CH_2-CH_3), 36.68 (C-4), 71.26 (C-6), 85.10 (C-2) ppm; EI-MS: m/z (%) = 159 (100), 103 (16), 87 (27), 69 (40), 57 (45), 56 (20).

5,5-Dimethyl-2,2-dibenzyl-1,3-oxathiane (**3**, $C_{20}H_{24}OS$)

Yield: 44%; colorless liquid; bp 64°C/1 mm Hg; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.93$ (s, 6H, 5-CH_3), 2.44 (d, $J = 1.7$ Hz, 2H, H-4), 3.03 (dd, $J = 14.3$, 1.7 Hz, 2H, 2-CH(H′)), 3.25 (dd, $J = 14.3$, 1.7 Hz, 2H, 2-CH(H′)), 3.62 (d, $J = 1.7$ Hz, 2H, H-6), 7.30–7.52 (overlapped peaks, 10H, aromatic protons) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 24.96$ (5-CH_3), 28.34 (C-5), 37.07 (C-4), 44.05 (CH_2), 71.97 (C-6), 85.05 (C-2), 126.54, 127.83, 130.95, 136.75 (aromatic carbon atoms) ppm; IR: $\bar{\nu} = 1068$, 1030, 799, 750, 699 cm^{-1}.

2-(2-Propyl)-5,5-dimethyl-1,3-oxathiane (**4**, $C_9H_{18}OS$)

Yield: 60%; colorless liquid; bp 56°C/2 mm Hg; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.84$ (s, 3H, 5-CH_3-*eq*), 0.98 (d, $J = 6.2$ Hz, 3H, CHCH_3(CH_3)), 1.01 (d, $J = 6.2$ Hz, 3H, $CHCH_3$(CH_3)), 1.20 (s, 3H, 5-CH_3-*ax*), 1.85 (heptet, $J = 6.2$ Hz, 1H, 2-CH(CH_3)$_2$), 2.39 (dd, $J = 13.3$, 2.2 Hz, 1H, H_{eq}-4), 2.84 (d, $J = 13.3$ Hz, 1H, H_{ax}-4), 3.25 (d, $J = 11.5$ Hz, 1H, H_{ax}-6), 3.59 (dd, $J = 11.5$, 2.2 Hz, 1H, H_{eq}-6), 4.42 (d, $J = 5.5$ Hz, 1H, H-2) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 18.73$ (2-CH(CH_3)$_2$), 23.01 (5-CH_3-*eq*), 26.53 (5-CH_3-*ax*), 28.32 (C-5), 33.54 (–CH(CH_3)$_2$), 40.14 (C-4), 80.06 (C-6), 89.43 (C-2) ppm; EI-MS: m/z (%) = 174 (14), 131 (88), 87 (11), 69 (100), 56 (23), 55 (20).

5,5-Dimethyl-2-phenyl-1,3-oxathiane (**5**, $C_{12}H_{16}OS$)

Yield: 48%; yellow crystals; mp 57.1–57.4°C; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.97$ (s, 3H, 5-CH_3-*eq*), 1.40 (s, 3H, 5-CH_3-*ax*), 2.47 (dd, $J = 13.4$, 2.3 Hz, 1H, H_{eq}-4), 3.20 (d, $J = 13.4$ Hz, 1H, H_{ax}-4), 3.49 (d, $J = 11.5$ Hz, 1H, H_{ax}-6), 3.78 (dd, $J = 11.5$, 2.3 Hz, 1H, H_{eq}-6), 5.66 (s, 1H, 2-H), 7.30–7.50 (m, 5H, aromatic protons) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 23.18$ (5-CH_3-*eq*), 26.66 (5-CH_3-*ax*), 28.12 (C-5), 41.63 (C-4), 80.25 (C-6), 84.59 (C-2), 126.34, 128.47, 128.59, 139.15 (aromatic carbon atoms) ppm; EI-MS: m/z (%) = 208 (100), 122 (24), 121 (23), 107 (27), 105 (34), 102 (96), 87 (46), 69 (90), 68 (54), 56 (74), 55 (30).

5,5-Dimethyl-2-(1-naphtyl)-1,3-oxathiane (**6**, $C_{16}H_{18}OS$)

Yield: 50%; white crystals; mp 150.9–151.1°C; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 1.00$ (s, 3H, 5-CH_3-*eq*), 1.44 (s, 3H, 5-CH_3-*ax*), 2.56 (dd, $J = 13.2$, 2.4 Hz, 1H, H_{eq}-4), 3.24 (d, $J = 13.2$ Hz, 1H, H_{ax}-4), 3.65 (d, $J = 11.5$ Hz, 1H, H_{ax}-6), 3.89 (dd, $J = 11.5$, 2.4 Hz, 1H, H_{eq}-6), 6.30 (s, 1H, H-2), 7.35–8.32 (overlapped peaks, 7H, aromatic protons) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 23.47$ (5-$CH_{3\text{-}eq}$), 26.71 (5-$CH_{3\text{-}ax}$), 28.44 (C-5), 41.88 (C-4), 80.80 (C-6), 82.61 (C-2), 123.41, 123.73, 124.66, 125.52, 125.74, 126.14, 128.79, 129.10, 133.74, 134.80 (aromatic carbon atoms) ppm; EI-MS: m/z (%) = 258 (100), 172 (17), 157 (17), 156 (89), 155 (43), 128 (22), 127 (32), 102 (16), 69 (12).

5,5-Dimethyl-2-(2-naphtyl)-1,3-oxathiane (**7**, $C_{16}H_{18}OS$)

Yield: 67%; white crystals; mp 121.8–122.7°C; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.97$ (s, 3H, 5-CH_3-*eq*), 1.40 (s, 3H, 5-CH_3-*ax*), 2.52 (dd, $J = 13.2$, 2.4 Hz, 1H, H_{eq}-4), 3.15 (d, $J = 13.2$ Hz, 1H, H_{ax}-4), 3.55 (d, $J = 11.7$ Hz, 1H, H_{ax}-6), 3.83 (dd, $J = 11.7$, 2.4 Hz, 1H, H_{eq}-6), 5.84 (s, 1H, H-2), 7.48–7.96 (overlapped peaks, 7H, aromatic protons) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 23.28$ (5-CH_3-*eq*), 26.68 (5-CH_3-*ax*), 28.19 (C-5), 41.71 (C-4), 80.30 (C-6), 84.70 (C-2), 124.23, 125.34, 125.77, 126.28,

127.78, 128.30, 133.19, 133.44, 136.60 (aromatic carbon atoms) ppm; EI-MS: m/z (%) = 258 (80), 172 (17), 171 (17), 157 (20), 156 (100), 155 (41), 129 (17), 128 (55), 127 (29), 102 (25), 69 (20), 56 (25).

2,5,5-Trimethyl-2-phenyl-1,3-oxathiane (**8**, $C_{13}H_{18}OS$)

Yield: 44%; colorless liquid; bp 110°C/1 mm Hg; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.78$ (s, 3H, 5-CH_3-*eq*), 1.28 (s, 3H, 5-CH_3-*ax*), 1.72 (s, 3H, 2-CH_3), 2.31 (dd, $J = 13.3$, 2.0 Hz, 1H, H_{eq}-4), 2.60 (d, $J = 13.3$ Hz, 1H, H_{ax}-4), 3.29 (d, $J = 11.9$ Hz, 1H, H_{ax}-6), 3.36 (dd, $J = 11.9$, 2.0 Hz, 1H, H_{eq}-6), 7.24–7.76 (overlapped peaks, 5H, aromatic protons) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 23.73$ (5-CH_3-*eq*), 26.14 (5-CH_3-*ax*), 27.68 (2-CH_3), 33.11 (C-5), 38.15 (C-4), 73.21 (C-6), 85.05 (C-2), 127.03, 127.61, 128.62, 143.33 (aromatic carbon atoms) ppm; EI-MS: m/z (%) = 222 (50), 207 (95), 121 (100), 105 (77), 102 (99), 87 (41), 77 (16), 66 (71), 65 (42), 56 (41), 55 (24), 51 (30).

2,5,5-Trimethyl-2-(p-nitrophenyl)-1,3-oxathiane (**9**, $C_{13}H_{17}O_3SN$)

Yield: 63%; white crystals; mp 119.6–120.2°C; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.81$ (s, 3H, 5-CH_3-*eq*), 1.26 (s, 3H, 5-CH_3-*ax*), 1.72 (s, 3H, 2-CH_3) 2.36 (dd, $J = 13.4$, 2.1 Hz, 1H, H_{eq}-4), 2.52 (d, $J = 13.4$ Hz, 1H, H_{ax}-4), 3.21 (d, $J = 12.1$ Hz, 1H, H_{ax}-6), 3.42 (dd, $J = 12.1$, 2.1 Hz, 1H, H_{eq}-6), 7.88 (d, $J = 8.9$ Hz, 2H, aromatic protons), 8.25 (d, $J = 8.9$ Hz, 2H, aromatic protons) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 23.62$ (5-CH_3-*eq*), 25.94 (5-CH_3-*ax*), 27.66 (2-CH_3), 32.52 (C-5), 38.01 (C-4), 73.43 (C-6), 84.14 (C-2), 123.91, 128.01, 147.41, 151.28 (aromatic carbon atoms) ppm; EI-MS: m/z (%) = 267 (25), 252 (43), 102 (100), 87 (37), 74 (21), 69 (85), 68 (54), 56 (66), 55 (22).

2,5,5-Trimethyl-2-(1-naphtyl)-1,3-oxathiane (**10**, $C_{17}H_{20}OS$)

Yield: 67%; yellow liquid; bp 40°C/0.4 mm Hg; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.67$ (s, 3H, 5-CH_3-*eq*), 1.36 (s, 3H, 5-CH_3-*ax*), 2.01 (s, 3H, 2-CH_3), 2.33 (dd, $J = 13.3$, 2.3 Hz, 1H, H_{eq}-4), 2.88 (d, $J = 13.3$ Hz, 1H, H_{ax}-4), 3.06 (d, $J = 11.5$ Hz, 1H, H_{ax}-6), 3.31 (dd, $J = 11.5$, 2.4 Hz, 1H, H_{eq}-6), 7.35–8.86 (overlapped peaks, 7H, aromatic protons) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 23.89$ (5-CH_3-*eq*), 26.36 (5-CH_3-*ax*), 27.31 (2-CH_3), 31.44 (C-5), 38.61 (C-4), 73.58 (C-6), 89.21 (C-2), 125.35, 125.55, 126.14, 126.40, 128.80, 129.30, 129.62, 131.23, 135.22, 136.98 (aromatic carbon atoms) ppm; IR: $\bar{\nu}$: 647, 725, 778, 805, 1026, 1050, 1108, 1132, 1173 cm^{-1}.

2,5,5-Trimethyl-2-(2-naphtyl)-1,3-oxathiane (**11**, $C_{17}H_{20}OS$)

Yield: 66%; colorless crystals; mp 67.3–68.4°C; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.75$ (s, 3H, 5-CH_3-*eq*), 1.31 (s, 3H, 5-CH_3-*ax*), 1.79 (s, 3H, 2-CH_3), 2.33 (dd, $J = 13.3$, 1.9 Hz, 1H, H_{eq}-4), 2.68 (d, $J = 13.3$ Hz, 1H, H_{ax}-4), 3.31 (d, $J = 11.9$ Hz, 1H, H_{ax}-6), 3.39 (dd, $J = 11.9$, 2.1 Hz, 1H, H_{eq}-6), 7.49–8.28 (overlapped peaks, 7H, aromatic protons) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 23.70$ (5-CH_3-*eq*), 26.20 (5-CH_3-*ax*), 27.70 (2-CH_3), 33.10 (C-5), 38.25 (C-4), 73.43 (C-6), 85.30 (C-2), 124.75, 125.64, 125.76, 126.26, 127.86, 128.43, 132.94, 133.45, 140.69 (aromatic carbon atoms) ppm; EI-MS: m/z (%) = 272 (51), 257 (38), 171 (41), 170 (60), 155 (100), 127 (37).

5,5-Dimethyl-2-ethyl-2-phenyl-1,3-oxathiane (**12**, $C_{14}H_{20}OS$)

Yield: 72%; colorless liquid; bp 112–115°C/2 mm Hg; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 0.77$ (s, 3H, 5-CH_3-*eq*), 0.80 (t, $J = 7.3$ Hz, 3H, 2-CH_2-C$\underline{H}_3$), 1.27 (s, 3H, 5-CH_3-*ax*), 1.99–2.03 (overlapped dq, 2H, 2-CH(H′)-CH_3), 2.32 (d, $J = 13.3$ Hz, 1H, 4-H_{eq}), 2.57 (d, $J = 13.3$ Hz, 1H, 4-H_{ax}), 3.32 (s, 2H, 6-H),

7.27–7.68 (m, 5H, aromatic protons) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 8.45$ ($-CH_2-\underline{C}H_3$), 23.76 (5-CH_3-*eq*), 26.19 (5-CH_3-*ax*), 27.99 (C-5), 37.73 ($-\underline{C}H_2-CH_3$), 38.14 (C-4), 72.94 (C-6), 89.01 (C-2), 127.52, 127.87, 128.39, 141.66 (aromatic carbon atoms) ppm; EI-MS: m/z (%) = 207 (95), 135 (16), 121 (12), 105 (58), 102 (12), 77 (31), 69 (17), 57 (19).

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