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# An Unexpected Synthesis of Novel Oxygen-Bridged 1,5-Benzothiazepine Derivatives and their Reductive Five-Membered-Ring Opening

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**Summary.** A convenient procedure is reported for the preparation of benzofuro-annelated 2-phenyl-1,5-benzothiazepine derivatives by oxidative cyclocondensation of phenolic $\beta$-diketones with *o*-aminothiophenol in *DMSO*. The regiochemistry of these compounds is proven by HMBC signals and the existence of a five-bond $^{19}F,^{13}C$-2 coupling. Surprisingly, treatment with $LiAlH_4$ at room temperature led to a double reduction under opening of the five-membered ring. Refluxing such solutions with a higher amount of $LiAlH_4$ gave rise to a further reduced derivative possessing the *trans*-configuration. All structures (regio- and stereochemistry) were assigned on the basis of NMR spectroscopic data.

**Keywords.** Benzofuro-annelated 1,5-benzothiazepines; 4,5-Dihydro-1,5-benzothiazepines; 2,3,4,5-Tetrahydro-1,5-benzothiazepines; Cyclocondensation; Regiochemistry; Stereochemistry; Conformational analysis.

## Introduction

Substituted benzothiazepines are known for their calcium antagonistic activity. A well-known example is diltiazem [1], an accepted drug for the treatment of cardiovascular disorders. During the last five years a number of compounds have been patented mostly in Japan and USA which have shown prominent calcium antagonistic activity. Mostly, they are derivatives of benzo-1,5-thiazepine-2-one bearing a substituted phenyl ring at C-2 [2] and alkoxycarbonyl or alkoxysulfonyl groups at C-3 [3]. The nitrogen atom at position 5 carries in most cases disubstituted aminoalkyl groups [4]. Oxygen-bridged sterically constrained heterocycles with complicated conformational behaviour are also gaining interest [5]. The syntheses of oxygen-bridged 1,5-benzothiazepine derivatives and dihydro analogues have been reported starting from chalcones [6] and from $\alpha,\beta$-enones [7].

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## Results and Discussion

Cyclocondensation reaction of some phenolic $\beta$-diketones (**1**) with *o*-aminothiophenol in *DMSO* has been reported to give 1,5-benzothiazepines with an *o*-hydroxyphenyl side chain (**2**) [8]. Adopting this procedure we attempted to react the phenolic $\beta$-diketones (**1a–1d**) [9] with *o*-aminothiophenol. Surprisingly, however, the cyclocondensation proceeded under oxidation to give oxygen-bridged 1,5-benzothiazepines **3a–d** in reasonable yields (Fig. 1), whereas the respective compounds **2a–d** were not detected. This offers a simple and effective synthetic pathway to this heterocyclic system.

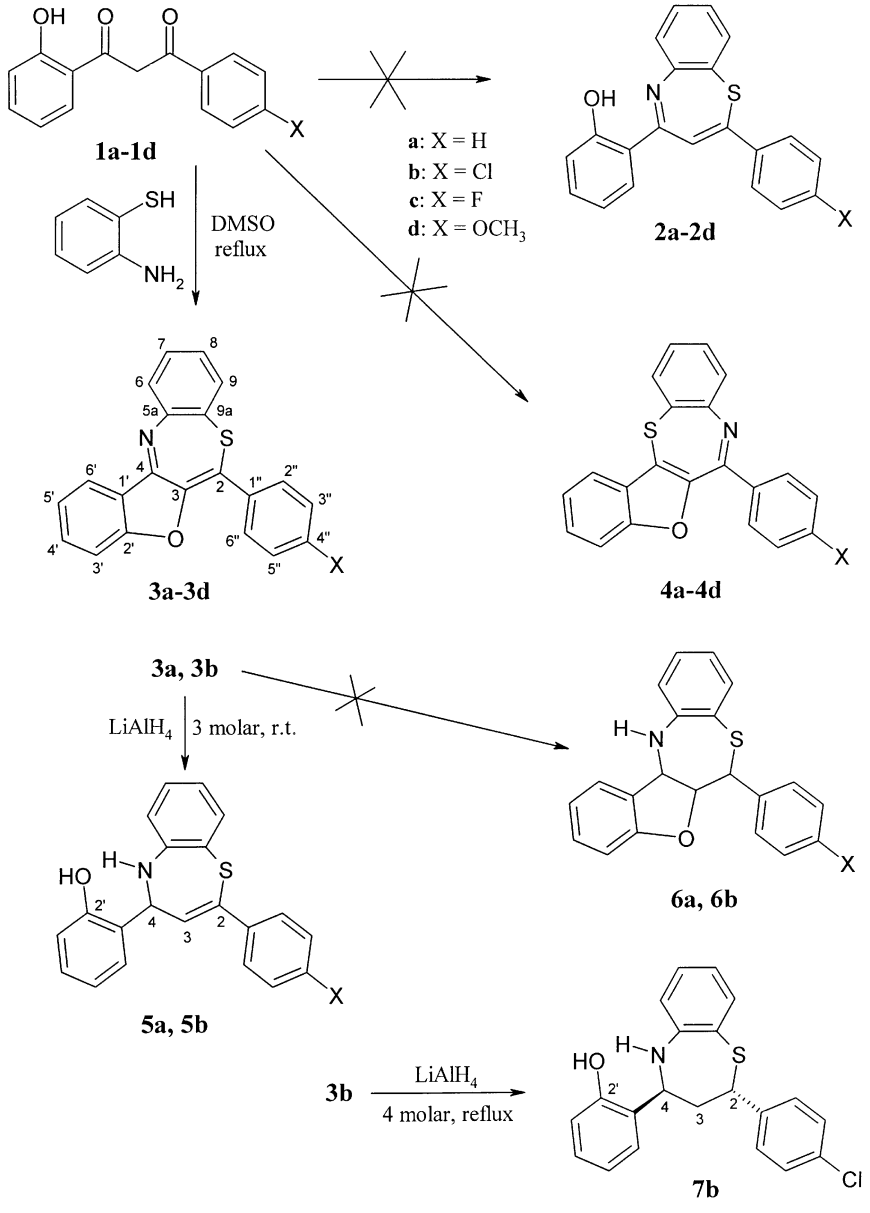



**Fig. 1.** Synthesis of compounds **3a–d**, **5a**, **b**, and **7a**, **b**

The structures of compounds **3a–d** were confirmed by spectroscopic techniques. The molecular ion peak in the mass spectrum of **3a** was observed at $m/z = 327$ instead of $m/z = 329$ for **2a**; the molecular formula based on the high resolution mass spectrum was found to be $C_{21}H_{13}NOS$ (and not $C_{21}H_{15}NOS$). The mass spectra of products **3b–d** showed analogous differences. Elemental analyses

**Table 1.** $^{1}H$ and $^{13}C$ chemical shifts (ppm) of compounds **3a–d**; in $CDCl_3$, relative to internal *TMS*; values in parentheses are coupling constants $^{n}J(^{19}F,^{1}H)$ or $^{n}J(^{19}F,^{13}C)$, respectively, in Hz

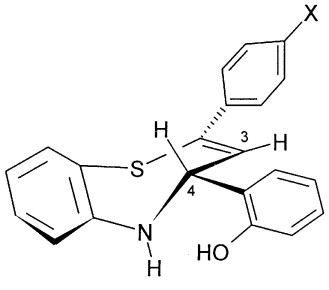
| | **3a** ($X = H$) | **3b** ($X = Cl$) | **3c** ($X = F$) | **3d** ($X = OCH_3$) |
|---|---|---|---|---|
| H-6 | 7.16 dd | 7.16 dd | 7.16 dd | 7.15 dd |
| H-7 | 7.01 td | 7.03 td | 7.02 td | 7.01 td |
| H-8 | 6.90 td | 6.92 td | 6.91 td | 6.90 td |
| H-9 | 6.74 dd | 6.76 dd | 6.75 dd | 6.75 dd |
| H-3′ | 7.09 dd | 7.08 dd | 7.09 dd | 7.09 dd |
| H-4′ | 7.43 td | 7.43 td | 7.43 td | 7.43 td |
| H-5′ | 7.25 td | 7.23 td | 7.25 td | 7.24 td |
| H-6′ | 8.25 dd | 8.23 dd | 8.24 dd | 8.24 dd |
| H-2″/6″ | 7.71 (AA′) | 7.68 (AA′) | 7.72 (AA′) (8.9) | 7.681 (AA′) |
| H-3″/5″ | 7.49 (XX′) | 7.44 (XX′) | 7.16 (XX′) (8.5) | 6.99 (XX′) |
| H-4″ | 7.47 (Y) | – | – | – |
| C-2 | 106.1 | 106.4 | 106.1 (0.9) | 105.2 |
| C-3 | 146.6 | 145.3 | 145.3 | 146.6 |
| C-4 | 148.7 | 148.4 | 148.9 | 148.9 |
| C-5a | 141.4 | 141.3 | 141.3 | 141.5 |
| C-6 | 128.9 | 129.1 | 129.0 | 128.8 |
| C-7 | 127.1 | 127.3 | 127.2 | 127.1 |
| C-8 | 127.0 | 127.1 | 127.1 | 126.9 |
| C-9 | 125.0 | 125.1 | 125.1 | 125.0 |
| C-9a | 121.1 | 120.8 | 120.8 | 121.2 |
| C-1′ | 120.4 | 120.3 | 120.3 | 120.4 |
| C-2′ | 154.5 | 154.4 | 154.4 | 154.5 |
| C-3′ | 117.0 | 117.0 | 117.0 | 117.0 |
| C-4′ | 132.0 | 130.9 | 132.1 | 132.0 |
| C-5′ | 124.9 | 125.1 | 125.0 | 124.8 |
| C-6′ | 125.0 | 125.1 | 125.1 | 125.0 |
| C-1″ | 132.5 | 132.1 | 128.6 (3.5) | 124.8 |
| C-2″/6″ | 128.0 | 128.8 | 130.2 (8.6) | 129.6 |
| C-3″/5″ | 128.5 | 129.4 | 115.7 (21.9) | 113.9 |
| C-4″ | 130.1 | 135.9 | 163.3 (251.3) | 160.8 |
| $OCH_3$ | – | – | – | 3.86 |
| $OCH_3$ | – | – | – | 55.4 |

also favour the structures **3**. Eight tertiary carbons instead of seven were observed in the $^{13}C$ NMR spectra. The assignment of the $^{1}H$ and $^{13}C$ NMR signals is based on extensive 2D spectroscopy (COSY, HMQC, and HMBC). The regiochemistry of the reaction, i.e. the fact that the products are of type **3** and not of type **4**, is unambiguously proven by two spectroscopic evidences: (*a*) there is a HMBC peak correlating C-4 with its high $\delta$-value (*ca.* 148.5 to 149 ppm; typical for C=N) to H-6′; (*b*) in **3c** a small but significant $^{19}F,^{13}C$ coupling of 0.9 Hz can be observed for the signal of C-2 at $\delta = 106.1$ ppm (typical for a $\beta$-carbon within an enol fragment). It should be noted that the compounds **3** differ from related ones reported earlier [5–7] which contain reduced atoms within the thiazepin ring.

Compounds **3a** and **3b** – as examples – were reduced by of $LiAlH_4$ (**3***M*) to yield **5a** and **5b** (Fig. 1). Although we expected the reduction of both double bonds (**6a** and **6b**), only the C=N double bond was reduced and, to our surprise, the oxygen bridge was opened as well. In the $^{13}C$ NMR spectrum of **5a** there is only one peak in the aliphatic region at $\delta = 61.2$ ppm (CH). The $^{1}H$ NMR spectrum shows two sharp signals at $\delta = 5.95$ and 4.97 ppm besides the signals in the aromatic and olefinic region. The proton at $\delta = 4.97$ ppm is attached to the carbon at $\delta = 61.2$ ppm as shown by the HMQC spectrum. This proves the presence of a -(H-4)-(C-4)-N- fragment. The hydrogen at $\delta = 5.95$ ppm is attached to the carbon at $\delta = 126.0$ ppm which is a typical $sp^2$-CH region. The assignment of this CH fragment to H-3/C-3 is proven by the HMBC spectrum correlating C-3 and H-4. H-4 showed a correlation (three-bond coupling) to the aromatic carbon carrying the OH group (C-2′). Finally, the $^{1}H$ NMR signals at $\delta = 8.30$ and 4.45 ppm are assigned to OH and NH protons. Analogous spectroscopic results were obatined for the chloro derivative **5b**.

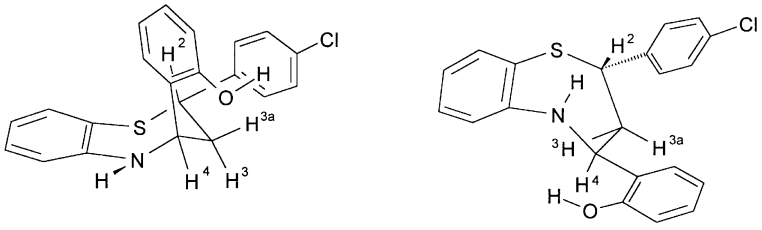
The suprisingly small vicinal coupling constant $^{3}J$(H-3,H-4) $= 1.3$ Hz indicates that the respective torsion angle (H-3/C-3/C-4/H-4) is close to 90°. This allows to define the preferred conformation of **5a** and **5b** as a half-chair with a *quasi*-equatorial phenol substituent at C-4 (Fig. 2).

In an attempt to achieve a fully reduced 2,3,4,5-tetrahydro-1,5-benzothiazepine derivative, a higher amount of $LiAlH_4$ was used for the reduction of **3b** as an example, and the reaction mixture was refluxed for 30 minutes after stirring overnight at room temperature. Thereby, a new reduction product **7b** (Fig. 1) was isolated. This compound contains an aliphatic fragment -NH-$C^4$H-$C^3H_2$-$C^2$H-S-




**5a:** $X$ = H
**5b:** $X$ = Cl

**Fig. 2.** Preferred conformation of **5a**, **b**




**A** (chair) **B** (twist-boat)

**Fig. 3.** Conformations of **7b**

**Table 2.** Torsion angles (°) of conformers **A** (chair) and **B** (twist-boat) of **7b**, calculated by the semiempirical AM1 method; experimental coupling constants $^3J_{H,H}$ (Hz) are given for comparison

| Fragment | **A** (chair) | **B** (twist-boat) | $^3J_{H,H}$/Hz |
|---|---|---|---|
| $C^{9a}$-S-$C^2$-$C^3$ | 76.3 | 25.9 | |
| S-$C^2$-$C^3$-$C^4$ | −78.0 | −83.6 | |
| $C^2$-$C^3$-$C^4$-N | 69.3 | 40.5 | |
| $C^{5a}$-N-$C^4$-$C^3$ | −73.1 | 54.2 | |
| $H^2$-$C^2$-$C^3$-$H^3$ | 165.4 | 156.9 | 12.0 |
| $H^2$-$C^2$-$C^3$-$H^{3a}$ | −76.5 | −86.2 | 3.1 |
| $H^4$-$C^4$-$C^3$-$H^3$ | 58.1 | 35.2 | 4.2 |
| $H^4$-$C^4$-$C^3$-$H^{3a}$ | −57.9 | −80.5 | 4.0 |
| $C^4$-$C^{1'}$-$C^{2'}$-O | 0.1 | −0.4 | |
| H-N-$C^4$-$H^4$ | 41.7 | 173.4 | – |
| S-$C^2$-$C^{1''}$-$C^{2''}$ | −66.8 | 94.8 | |

which could be identified from 1D and 2D NMR spectra. The corresponding $^1$H,$^1$H coupling constants are: $^2J$(H-3,H-3a) = 15.3 Hz, $^3J$(H-2,H-3) = 12.0 Hz, $^3J$(H-2,H-3a) = 3.1 Hz, $^3J$(H-3,H-4) = 4.2 Hz and $^3J$(H-3a,H-4 = 4.0 Hz The most diagnostic one of them is $^3J$(H-2,H-3) = 12.0 Hz clearly indicates the *anti*-periplanar orientation of the hydrogens involved. There are only two conceivable conformations of the *trans*-configurated **7b** – the *cis*-configuration can be excluded – which fit to this combination of couplings, structures **A** (chair) and **B** (twist-boat), as depicted in Fig. 3.

Semiempirical calculations (AM1) of **7b** slightly favour the twist-boat conformation **B**, but the difference of ca 1 kJ/mol is possibly within the error limits of the calculation. If, however, the geometry of the calculated structures (Table 2) and the experimental vicinal $^1$H,$^1$H coupling constants are evaluated using the *Karplus* equation, the chair conformation is more plausible. Especially, the coupling constant of 4.0 Hz for the $H^4$-$C^4$-$C^3$-$H^{3a}$ fragment fits much better to the chair (torsion angle = −58°) than to the half-boat conformation (torsion angle = −81°).

## Experimental

Melting points were determined on a Gallenkamp digital melting point apparatus and are uncorrected. IR spectra were recorded on a Hitachi 270-50 IR spectrophotometer. $^1$H and $^{13}$C NMR

spectra were recorded on a Bruker AM-250 (**3a–d**) and a Bruker DRX-400 spectrometer (**5a**, **b**, **7b**) using $CDCl_3$ as solvent. Bruker standard software was employed for the multipulse experiments (DEPT, COSY, HMQC, HMBC). Electron-impact mass spectra were recorded on Varian Mat CH-5. UV spectra were recorded on a Shimadzu UV-265 spectrophotometer. Column chromatography was performed on silica gel (60–120 mesh; Merck).

*General procedure for the cyclocondensation of β-diketones with o-aminothiophenol*

1-(2′-Hydroxyphenyl)-3-(phenyl)- or the respective -(*p*-substituted phenyl)-1,3-propanediones (**1a–d**) were refluxed with *o*-aminothiophenol using *DMSO* as solvent. After 5 h the mixture was poured onto crushed ice and extracted with ethylacetate. The organic layer was dried (anhydrous $Na_2SO_4$) and evaporated *in vacuo*. The solid obtained was chromatographed on a silica gel column using benzene as eluent. The compounds were recrystallized from absolute ethanol.

*Benzofuro-[1′,2′-c]-2-phenyl-1,5-benzothiazepine* (**3a**; $C_{21}H_{13}NOS$)

Yield: 69.7%. m.p.: = 166–167°C; IR (KBr): $\tilde{\nu} = 3982$, 3940, 3652, 3544, 3334, 3250, 3196, 3034, 2848, 2680, 2260, 2128, 2026, 1962, 1938, 1896, 1839, 1602, 1530, 1488, 1458, 1446, 1347, 1290, 1260, 1221, 1134, 1056, 1020, 915, 741, 681 $cm^{-1}$; UV (MeOH): $\lambda_{max}(\varepsilon) = 511$ (56925), 323 (20780), 286 (21080), 253 (31300) nm; for NMR data, see Table 1; EI-MS: *m/z* (%) = 327 ($M^+$, 100), 326 (8), 299 (9), 297 (8), 295 (13), 267 (4), 222 (4), 164 (8), 148 (9).

*Benzofuro-[1′,2′-c]-7-(4′′-chlorophenyl)-1,5-benzothiazepine* (**3b**; $C_{21}H_{12}ClNOS$)

Yield: 73.4%. m. p.: 182–183.5°C; IR (KBr): $\bar{\nu} = 3970$, 3844, 3778, 3670, 3448, 3412, 3190, 3040, 2914, 2800, 2668, 2284, 2146, 1983, 1896, 1602, 1533, 1482, 1461, 1395, 1344, 1290, 1260, 1224, 1134, 1083, 1062, 1014, 738, 717 $cm^{-1}$; UV (MeOH): $\lambda_{max}(\varepsilon) = 511$ (50615), 328 (17380), 287 (17760), 252 (24550) nm; for NMR data, see Table 1; EI-MS: *m/z* (%) = 361 ($M^+$, 100), 360 (7), 333 (8), 297 (10), 329 (8), 250 (10), 222 (6), 164 (5), 148 (23).

*Benzofuro-[1′,2′-c]-2-(4′′-fluorophenyl)-1,5-benzothiazepine* (**3c**; $C_{21}H_{12}FNOS$)

Yield: 64.1%; m.p.: 188.1–189°C; IR (KBr): $\nu = 3796$, 3658, 3262, 3046, 2908, 2842, 2254, 2152, 2086, 1893, 1602, 1536, 1497, 1461, 1404, 1344, 1266, 1218, 1155, 1137, 1077, 1056, 1014, 741 $cm^{-1}$; UV (MeOH): $\lambda_{max}(\varepsilon) = 508$ (50535), 324 (17000), 288 (16920), 251 (25195) nm; for NMR data, see Table 1; EI-MS: *m/z* (%) = 345 ($M^+$, 100), 344 (9), 317 (10), 313 (10), 285 (4), 250 (6), 222 (4), 148 (4).

*Benzofuro-[1′,2′-c]-2-(4′′-methoxyphenyl)-1,5-benzothiazepine* (**3d**; $C_{22}H_{15}NO_2S$)

Yield: 72.5%; m.p.: 150.8–152.5°C; IR (KBr): $\tilde{\nu} = 3916$, 3604, 3472, 3040, 2914, 2830, 2056, 1929, 1899, 1605, 1533, 1500, 1448, 1347, 1302, 1239, 1173, 1137, 1104, 1059, 1023, 930, 813, 744 $cm^{-1}$; UV (MeOH): $\lambda_{max}(\varepsilon) = 517$ (68530), 353 (22310), 297 (19200), 252 (32235) nm; for NMR data, see Table 1; EI-MS: *m/z* (%) = 357 ($M^+$, 100), 342 (10), 314 (10), 282 (10), 178 (21), 164 (3).

*Syntheses of* **5a** and **5b**

To a stirred suspension of 3 mmol $LiAlH_4$ in 30 $m^3$ dry *THF*, a solution of 1 mmol **3a** or **3b** in *THF* was added dropwise. The reaction mixture was stirred thoroughly and monitored by TLC (petroleum

ether:ethylacetate = 4:1). After stirring overnight at room temperature the reaction mixture was cooled on ice. Water was added dropwise to destroy $LiAlH_4$, followed by addition of 10% $H_2SO_4$ until the solution was clear. *THF* was evaporated *in vacuo*, and the remaining aqueous solution was extracted with ethyl acetate. The organic layer was dried over anhydrous $Na_2SO_4$ and evaporated to dryness. The residue was purified by column chromatography using petroleum ether and ethylaceta te (7:1).

*2-Phenyl-4-(2′-hydroxyphenyl)-4,5-dihydro-1,5-benzothiazepine* (**5a**; $C_{21}H_{17}NOS$)

Yield: 39%; m.p.: 120°C; $^1$H NMR ($CDCl_3$, $\delta$, 400 MHz): 4.45 (bs, 1H, NH), 4.97 (H-4, d, $J = 1.3$ Hz), 5.92 (H-3, d, $J = 1.3$ Hz), 6.68 (H-3′, d), 6.85 (H-8, dd), 6.85 (H-4′, dd), 6.92 (H-6, d), 6.97 (H-5′, dd), 7.09 (H-6′, d), 7.12 (H-9, d), 7.16 (H-4″, dd), 7.24 (H-7, dd), 7.28 (H-3″/5″, dd), 7.34 (H-2″/6″, d), 8.30 (bs, 1H, OH) ppm; $^{13}$C NMR ($CDCl_3$, $\delta$, 100.6 MHz): 61.2 (C-4), 117.6 (C-6), 118.6 (C-3′), 120.3 (C-8), 120.4 (C-1′), 122.6 (C-4′), 123.1 (C-9a), 123.8 (C-3), 126.0 (C-5′), 126.3 (C-6′), 127.5 (C-4″), 128.4 (C-3″/5″), 129.1 (C-2″/6″), 130.4 (C-9), 130.6 (C-7), 131.4 (C-2), 135.6 (C-1″), 141.1 (C-5a), 155.8 (C-2′) ppm; EI-MS: *m/z* (%) = 331 ($M^+$, 70), 298 (5), 254 (16), 240 (100), 228 (22), 207 (38), 196 (13), 178 (16), 165 (6), 149 (7), 136 (15), 122 (9), 109 (11), 91 (9), 77 (18).

*2-(4″-Chlorophenyl)-4-(2′-hydroxyphenyl)-4,5-dihydro-benzothiazepine* (**5b**; $C_{21}H_{16}ClNOS$)

Yield: 45%; m.p.: 156°C; $^1$H NMR ($CDCl_3$, $\delta$, 400 MHz): 4.5 (broad, 1H, NH), 4.97 (H-4, d, $J = 1.3$ Hz), 5.92 (H-3, d, $J = 1.3$ Hz), 6.68 (H-3′, d), 6.91 (H-4′, dd), 6.91 (H-8, dd), 6.97 (H-6, d), 7.02 (H-5′, dd), 7.15 (H-6′, d), 7.19 (H-9, d), 7.25 (H-2″/6″, d), 7.29 (H-7, dd), 7.30 (H-3″/5″, dd), *ca.* 7.8 (broad, 1H, OH) ppm; $^{13}$C NMR ($CDCl_3$, $\delta$, 100.6 MHz): 61.5 (C-4), 117.6 (C-6), 118.3 (C-3′), 120.2 (C-8), 120.2 (C-1′), 122.6 (C-4′), 123.4 (C-3 and C-9a), 126.1 (C-5′), 126.3 (C-6′), 134.4 (C-4″), 129.1 (C-3″/5″), 130.2 (C-9), 130.4 (C-2″/6″), 130.5 (C-7), 132.9 (C-2), 134.4 (C-1″), 141.6 (C-5a), 155.9 (C-2′) ppm; EI-MS: *m/z* (%) = 365/367 ($M^+$+32/10), 331 (30), 272/274 (11/3), 254 (16), 240 (100), 227 (29), 207 (60), 178 (38), 149 (21), 136 (48), 122 (34), 109 (27), 97 (38), 71 (38), 207 (38), 196 (13), 165 (6), 91 (9), 77 (18), 69 (45), 57 (47).

*trans-2-(4″-Chlorophenyl)-4-(2′-hydroxyphenyl)-2,3,4,5-tetrahydro-1,5-benzothiazepine* (**7b**; $C_{21}H_{18}ClNOS$)

The synthetic procedure was analogous to those for **5a** and **5b** except for a higher amount of LiAlH4 employed (4 mmol) and the fact that after overnight stirring at room temperature the reaction mixture was refluxed for 30 min before work-up.

Yield: 65%; m.p.: 170°C; IR (KBr) $\tilde{\nu} = 3350$ (br, NH/OH), 3044, 2944, 1584, 1488, 1456, 1248, 1092, 1012, 824, 752 $cm^{-1}$; $^1$H NMR ($CDCl_3$, $\delta$, 400 MHz): 2.22 (H-3a, ddd), 2.48 (H-3, ddd), 4.1 (NH), 4.39 (H-2, dd), 4.81 (H-4, dd), 6.88 (H-5′, td), 7.01 (H-4′, td), 7.01 (H-3′, dd), 7.04 (H-6′, dd), 7.15 (H-8, td), 7.15 (H-2″/6″, m), 7.24 (H-3″/5″, m), 7.28 (H-6, dm), 7.31 (H-7, td), 7.57 (H-9, dd), 10.1 (OH) ppm; $^{13}$C NMR ($CDCl^3$, $\delta$, 100.6 MHz): 118.1 (C-3′), 120.3 (C-5′), 122.5 (C-6), 125.3 (C-8) 125.5 (C-1′), 126.4 (C-9a), 128.7 (C-2″/6″), 128.8 (C-6′), 129.2 (C-3″/5″), 129.7 and 129.8 (C-7 and C-4′), 133.2 (C-4″), 134.2 (C-9), 144.0 (C-1″), 146.4 (C-5a), 159.6 (C-2′) ppm; EI-MS: *m/z* (%) = 368/370 ($M^+$, 31/13), 334/336 (32/11), 243 (41), 229 (40), 228 (100), 149 (7), 136 (73), 125 (12), 109 (14), 77 (8).

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## References

[1] Kurokawa M, Sato F, Honda Y, Uno H (1991) J Med Chem **34**: 593
[2] Bousquet A, Charpiol A (1991) Eur Pat Appl Ep 381, 570; Chem Abstr (1991) **114**: 102070n
[3] Takatani M (1987) Jpn Kobai Tokkyo Koho JP 61, 130, 277; Chem Abstr (1987) **106**: 50265t
[4] Mohecsi E, O'Brien PJ, US 4, 912, 102; Chem Abstr (1990) **113**: 172061p
[5] Svetlik J, Turecek F (1988) J Chem Soc Perkin Trans 2053
[6] Svetlik J, Hanus V, Bella J (1989) Liebigs Ann Chem 91
[7] Tóth G, Szöllösy Á, Lévai A, Duddeck H (1982) Org Magn Reson **20**: 133
[8] Umesh CP, Gaur BS, Kumar V (1986) J Indian Chem Soc **63**: 680
[9] Ahmad R, Malik MA, Zia-ul-Haq M (1990) J Chem Soc Pak **12**: 352

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