Oxazepines and thiazepines 46.¹ Synthesis of tetracyclic 1,5-benzothiazepines by the reaction of α,β,γ,δ-unsaturated ketones with 2-aminothiophenol

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Dedicated to Professor Dr. Kálmán Hideg on the occasion of his 75th birthday

Abstract

Hitherto unknown tetracyclic 1,5-benzothiazepines **9-16** have been synthesized by an acidcatalyzed reaction of exocyclic $\alpha,\beta,\gamma,\delta$ -unsaturated ketones **1-8** and 2-aminothiophenol. Structures of all new compounds have been elucidated by microanalyses, ¹H-, ¹³C-NMR, IR and mass spectroscopic measurements.

Keywords: 2-Aminothiophenol, 3-cinnamylidenechromanones, 3-cinnamylidene-1-thiochromanones, tetracyclic 1,5-benzothiazepines

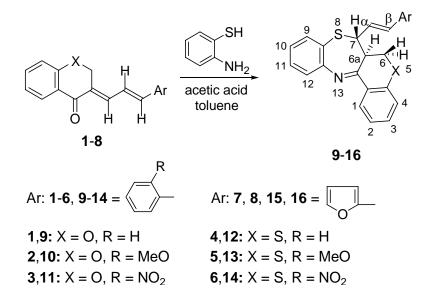
Introduction

The 1,5-benzothiazepines are important nitrogen- and sulfur-containing seven-membered heterocyclic compounds in drug research since they possess diverse bioactivities.²⁻⁶ As a result, various procedures have been worked out for their synthesis and numerous derivatives have been published in the literature. Probably owing to their easy availability and bioactivities, a well known group comprises the 2,3-dihydro-1,5-benzothiazepines. The reaction of α , β -unsaturated ketones and 2-aminothiophenol is an especially convenient and versatile method for their preparation.⁷⁻¹⁷ Reaction of exocyclic α , β -unsaturated ketones with 2-aminothiophenol afforded related tetracyclic 1,5-benzothiazepines.¹⁸⁻²⁷ However, for the synthesis of 1,5-benzothiazepines by the reaction of the related α , β , γ , δ -unsaturated ketones with 2-aminothiophenol only one example has been described in the literature.²⁸ We have synthesized 4-aryl-2,3-dihydro-2-styryl-1,5-benzothiazepines as sole products in this way. Reaction of exocyclic α , β , γ , δ -unsaturated

ketones and 2-aminothiophenol has not hitherto been published. For this reason, as a continuation of our study on the reaction of exocyclic α,β -unsaturated ketones with 2-aminothiophenol, in our present paper we report on the first synthesis of tetracylic 1,5-benzothiazepines by the reaction of exocyclic $\alpha,\beta,\gamma,\delta$ -unsaturated ketones and 2-aminothiophenol under acid-catalyzed conditions.

Results and Discussion

Studying the synthesis of the related 4-aryl-2,3-dihydro-2-styryl-1,5-benzothiazepines²⁸ we have investigated various conditions for the reaction of $\alpha,\beta,\gamma,\delta$ -unsaturated ketones (*E*,*E*-cinnamylideneacetophenones) with 2-aminothiophenol to afford 1,5-benzothiazepines. Slightly acidic reaction conditions proved to be the most convenient for this purpose.²⁸



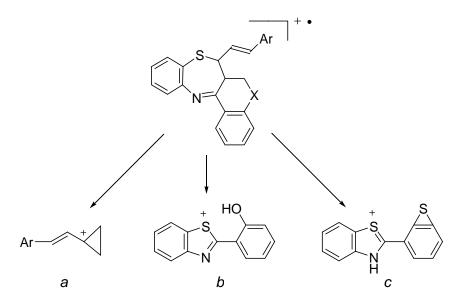
Scheme 1

Utilizing this experience, exocyclic $\alpha,\beta,\gamma,\delta$ -unsaturated ketones **1-8** were allowed to react with 2-aminothiophenol in a mixture of boiling toluene and acetic acid and tetracyclic 1,5-benzothiazepines **9-16** (Scheme 1) were obtained in relatively good or medium yields (59-71%, *cf.* Experimental Section). Neither the yield nor the course of the reaction were influenced by the presence of an electron donor or an electron acceptor *o*-substituent in the starting material. Replacement of the styryl group by a 2-(furan-2-yl)ethenyl one slightly enhaced the yield of the formation of 1,5-benzothiazepines **15** and **16**.

Structures of all new compounds have been elucidated by microanalyses, IR, ¹H-, ¹³C-NMR and mass spectroscopic measurements. Elemental analyses unambiguously proved the elemental composition of all new compounds which was confirmed by their mass spectra. In their IR

spectra a characteristic C=N band was assigned aroud 1600 cm⁻¹ referring to the presence of a C=N double bond in the seven-membered heterocycle. ¹H-NMR chemical shifts, multiplicity and coupling constant values (*cf.* Experimental Section) of protons attached to the C-6, C-6a and C-7 carbon atoms unequivocally prove the tetracyclic 6a,7-dihydro-1,5-benzothiazepine structure. Coupling constant values of 11-12 Hz indicate that the 6a-H and 7-H protons are *trans*-oriented as in the case of all the related tetracyclic 6a,7-dihydro-1,5-benzothiazepines.^{18-20,22,25} This similarity also indicates a stereoselective formation of one diastereomer compounds **9-16** depicted in Scheme 1. ¹³C-NMR chemical shift values of the aliphatic carbon atoms C-6 (*ca.* 66 ppm for the benzopyrano derivatives **9-11** and **15** and *approx.* 29 ppm for the 1-benzothiopyrano compounds **12-14** and **16**), C-6a (40-42 ppm) and C-7 (58-59 ppm) corroborate the tetracyclic 6a,7-dihydro-1,5-benzothiazepine structure deduced from the ¹H-NMR spectroscopic data. These ¹³C-NMR chemical shift values are in harmony with those values of the related tetracyclic 6a,7-dihydro-1,5-benzothiazepines.¹⁸⁻²⁰

A weak but distinct molecular ion was detected in the EI 70 eV mass spectra of the tetracyclic benzothiazepines 9-16. In the course of their fragmentation, these benzothiazepine molecules are split into two major fragment ions, *viz.* ions *a*, *b* or *c* (Scheme 2). Fragment ion *a* was detected in each spectra which correspond to the base peak in the case of benzopyrano derivatives 9-11 and 15. Fragment ion *b* appears at m/z 227 in the mass spectra of compounds 9-11 and 15. However, in the mass spectra of the benzothiopyrano derivatives 12-14 and 16 the base peak is the fragment ion *c* at m/z 242. This fragmentation pattern unequivocally proves the structures of tetracyclic benzothiazepines 9-16 elucidated by other spectroscopic methods.



Scheme 2

It is well known that the formation of 1,5-benzothiazepines by the reaction of α,β unsaturated ketones and 2-aminothiophenol is a two-step process.²⁷ The first step is the Michael addition of the mercapto group to the β -carbon atom of the unsaturated ketone. The second step is the ring closure of the Michael adduct to form the seven-membered heterocycle. In some cases, the Michael adduct can be isolated and then converted into the appropriate 1,5benzothiazepine on an acid-catalyzed cyclization.²⁷ In our previous studies on the preparation of tetracylic 1,5-benzothiazepines by the reaction of exocyclic α , β -unsaturated ketones with 2aminothiophenol¹⁸ we isolated Michael adducts and determined their stereochemistry. As expected, owing to the formation of two centres of chirality, these adducts proved to be diastereomeric mixtures. However, an acid-catalyzed ring closure of these mixtures afforded only one diastereomer of the appropriate tetracyclic 1,5-benzothiazepines stereochemistry of which was equal to that of one of the two diastereomers of the Michael adducts. It means that the "stereochemically unfavoured" distereomer gave tetracyclic 1,5-benzothiazepine on an acidcatalyzed epimerization followed by cyclization.

Although in our present study on the reaction of $\alpha, \beta, \gamma, \delta$ -unsaturated ketones and 2aminothiophenol no Michael adduct could be isolated, a similar reaction mechanism can be supposed resulting in high stereoselectivity. On the basis of our previous¹⁸ and present experimental results, it can be concluded that the acid-catalyzed reaction of these two groups of unsaturated ketones with 2-aminothiophenol leads to the formation of the thermodynamically more stable diastereomer of tetracyclic 1,5-benzothiazepines as sole isolable products.

In conclusion, we have conducted the first, stereoselective synthesis of hitherto unknown tetracyclic 6a,7-dihydro-7-styryl-1,5-benzothiazepines by an acid-catalyzed reaction of exocyclic $\alpha,\beta,\gamma,\delta$ -unsaturated ketones and 2-aminothiophenol. Our experimental results also prove that the site of the Michael addition of the thiol group is the β -carbon atom of the starting $\alpha,\beta,\gamma,\delta$ -unsaturated ketones as in the cases of the reaction of such unsaturated ketones with dinucleophiles.²⁸⁻³¹

Experimental Section

General Procedures. Melting points were determined on a Kofler hot-stage apparatus and are uncorrected. ¹H- and ¹³C-NMR spectra were measured with Bruker WP 200 SY and Bruker Avance DRX 500 spectrometers at 200/50 and 500/125 MHz in CDCl₃ (internal standard TMS, $\delta = 0.0$ ppm). The IR spectra were obtained in KBr discs with a Perkin-Elmer 16 PC instrument. Mass spectra were recorded on a VG Trio-2 apparatus. Elemantal analyses (C, H, N) were measured in-house with a Carlo Erba 1106 EA instrument. Thin layer chromatography (TLC) was performed on Kieselgel 60 F₂₅₄ (Merck) layer and the column chromatography on Kieselgel 60 columns using toluene:ethyl acetate (4:1 v/v) as eluent.

General method for the synthesis of benzothiazepines 9-16

A mixture of exocyclic $\alpha,\beta,\gamma,\delta$ -unsaturated ketone (1-8, 5.0 mmoles), 2-aminothiophenol (6.0 mmoles), acetic acid (5 mL) and toluene (50 mL) was heated at reflux for 5 h, then the solvent

was evaporated under reduced pressure. The benzothiazepines (9-16, Scheme 1) obtained were purified by column chromatography as indicated above.

6a,7-Dihydro-7-styryl-6H-benzopyrano[**3,4-c**][**1,5**]**benzothiazepine** (**9**). Isolated as white needles in 67% yield, mp 174-175 °C; ¹H-NMR (δ, CDCl₃): 2.60 (1H, m, 6a-H), 4.20 (1H, dd, J = 9.4, 12.2 Hz, 6-H), 4.41 (1H, dd, J = 5.9, 12.2 Hz, 6-H), 4.62 (1H, t, J = 11.2, 7-H), 6.16 (1H, dd, J = 13.1, 15.0 Hz, α-H), 6.52 (1H, d, J = 15.0 Hz, β-H), 7.01-8.30 (m, 13 arom. H); ¹³C-NMR (δ, CDCl₃): 42.0, 58.7, 65.7, 106.4, 117.6, 120.1, 121.4, 122.8, 124.9, 125.2, 126.6, 128.3, 128.6, 130.0, 131.1, 133.3, 135.5, 136.1, 151.7, 158.0, 162.9; IR (cm⁻¹): 1601, 1578, 1452, 1320, 1262, 1210, 1129, 1061, 968, 813, 754, 691; MS (m/z): 369 (M⁺, 6), 338 (4), 227 (8), 143 (100); Anal. Calcd. for C₂₄H₁₉NOS: C, 78.03; H, 5.18; N, 3.79. Found: C, 78.12; H, 5.23; N, 3.82.

6a,7-Dihydro-7-(2-methoxystyryl)-6*H***-benzopyrano[3,4-c][1,5]benzothiazepine (10).** Isolated as pale yellow needles in 66% yield, mp 154-155 °C; ¹H-NMR (δ, CDCl₃): 2.42 (1H, m, 6a-H), 3.74 (3H, s, MeO), 3.87 (1H, dd, J = 11.7, 14.8 Hz, 6-H), 4.28 (1H, dd, J = 8.7, 14.8 Hz, 6-H), 4.51 (1H, dd, J = 8.1, 11.3 Hz, 7-H), 6.26 (1H, dd, J = 9.4, 15.7 Hz, α-H), 6.83 (1H, d, J = 15.7, β-H), 6.89-8.36 (m, 12 arom, H); ¹³C-NMR (δ, CDCl₃): 42.2, 55.4, 59.2, 66.8, 110.8, 117.5, 120.4, 121.9, 122.9, 124.8, 125.0, 125.8, 126.7, 128.9, 133.2, 135.5, 145.0, 151.6, 156.9, 158.0, 165.0; IR (cm⁻¹): 1600, 1576, 1464, 1320, 1245, 1212, 1129, 1029, 975, 814, 752; MS (m/z): 399 (M⁺, 8), 278 (7), 227 (13), 173 (100); Anal. Calcd. for C₂₅H₂₁NO₂S: C, 75.17; H, 5.30; N, 3.50. Found: C, 75.07; H, 5.36; N, 3.45.

6a,7-Dihydro-7-(2-nitrostyryl)-6*H***-benzopyrano[3,4-c][1,5]benzothiazepine (11).** Isolated as yellow plates in 61% yield, mp 122-123 °C; ¹H-NMR (δ, CDCl₃): 2.73 (1H, m, 6a-H), 4.16 (1H, dd, J = 11.3, 14.5 Hz, 6-H), 4.33 (1H, dd, J = 8.4, 14.5 Hz, 6-H), 4.57 (1H, dd, J = 9.5, 11.7 Hz, 7-H), 6.18 (1H, dd, J = 9.5, 15.3 Hz, α-H), 7.01 (1H, d, J = 15.3 Hz, β-H), 7.06-8.32 (m, 12 arom. H); ¹³C-NMR (δ, CDCl₃): 41.8, 57.7, 66.6, 117.7, 119.9, 121.9, 122.3, 124.8, 125.0, 126.6. 128.8, 130.1, 132.1, 133.0, 133.4, 135.4, 151.7, 158.0, 162.8; IR (cm⁻¹): 1602, 1578, 1522, 1479, 1452, 1344, 1320, 1262, 1211, 1129, 962, 864, 749; MS (m/z): 414 (M⁺, 1), 227 (52), 198 (17), 156 (100); Anal. Calcd. for C₂₄H₁₈N₂O₃S: C, 69.56; H, 4.38; N, 6.76. Found: C, 69.66; H, 4.44; N, 6.72.

6a,7-Dihydro-7-styryl-6H-[1]benzothiopyrano[3,4-c][1,5]benzothiazepine (12). Isolated as pale yellow needles in 59% yield, mp 186-187 °C; ¹H-NMR (δ, CDCl₃): 2.80 (1H, m, 6a-H), 3.06 (1H, dd, J = 9.8, 13.4 Hz, 6-H), 3.37 (1H, dd, J = 7.6, 13.4 Hz, 6-H), 4.81 (1H, t, J = 11.6 Hz, 7-H), 6.27 (1H, t, J = 15.8 Hz, α-H), 6.53 (1H, d, J = 15.8 Hz, β-H), 7.10-8.56 (m, 13 arom. H); ¹³C-NMR (δ, CDCl₃): 29.2, 40.5, 58.9, 118.1, 122.1, 123.4, 124.8, 125.3, 126.6, 127.9, 128.2, 128.6, 129.9, 130.7, 131.0, 132.6, 135.3, 136.2, 137.3, 145.0, 151.6, 154.5; IR (cm⁻¹): 1600, 1575, 1448, 1315, 1188, 1157, 1072, 961, 888, 757, 731, 690; MS (m/z): 385 (M⁺, 4), 338 (6), 294 (4), 242 (100); Anal. Calcd. for C₂₄H₁₉NS₂: C, 74.79; H, 4.97; N, 3.63. Found: C, 74.86; H, 4.92; N, 3.69.

6a,7-Dihydro-7-(2-methoxystyryl)-6*H***-[1]benzothiopyrano**[**3,4-c**][**1,5]benzothiazepine** (**13**). Isolated as yellow needles in 61% yield, mp 178-179 °C; ¹H-NMR (δ , CDCl₃): 2.78 (1H, m, 6a-

H), 3.12 (1H, dd, J = 8.9, 14.0 Hz, 6-H), 3.41 (1H, dd, J = 7.1, 14.0 Hz, 6-H), 3.84 (3H, s, MeO), 4.84 (t, J = 12.1 Hz, 7-H), 6.22 (1H, dd, J = 9.8, 15.8 Hz, α-H), 6.80 (1H, d, J = 15.8 Hz, β-H), 6.93-8.60 (m, 12 arom. H); ¹³C-NMR (δ, CDCl₃): 29.3, 40.7, 55.4, 59.5, 110.9, 120.5, 123.5, 124.7, 125.2, 126.8, 127.7, 128.8, 129.9, 130.9, 135.2, 137.3, 151.5, 156.9, 164.5; IR (cm⁻¹): 1598, 1576, 1488, 1462, 1315, 1291, 1115, 1033, 967, 886, 751; MS (m/z): 415 (M⁺, 2), 368 (4), 294 (5), 242 (100); Anal. Calcd. for $C_{25}H_{21}NOS_2$: C, 72.28; H, 5.09; N, 3.37. Found: C, 72.20; H, 5.15; N, 3.41.

6a,7-Dihydro-7-(2-nitrostyryl)-6H-[1]benzothiopyrano[3,4-c][1,5]benzothiazepine (14). Isdolated as yellow plates in 63% yield, mp 181-182 °C; ¹H-NMR (δ, CDCl₃): 2.84 (1H, m, 6a-H), 3.08 (1H, dd, J = 9.1, 14.3 Hz, 6-H), 3.36 (1H, dd, J = 7.4, 14.3 Hz, 6-H), 4.92 (1H, dd, J = 9.8, 11.3 Hz, 7-H), 6.17 (1H, dd, J = 9.7, 15.6 Hz, α-H), 7.01 (1H, d, J = 15.6 Hz, β-H), 7.10-8.62 (m, 12 arom. H); ¹³C-NMR (δ, CDCl₃): 29.1, 40.4, 58.0, 122.9, 124.6, 124.8, 125.2, 126.3, 127.8, 129.0, 130.0, 131.1, 132.9, 133.1, 135.0, 137.4, 147.6, 151.6, 164.4; IR (cm⁻¹): 1602, 1577, 1518, 1345, 1313, 963, 759, 690; MS (m/z): 430 (M⁺, 1), 383 (8), 242 (100), 156 (14); Anal. Calcd. for C₂₄H₁₈N₂O₂S₂: C, 66.97; H, 4.21; N, 6.50. Found: C, 67.0.7; H, 4.24; N, 6.57.

6a,7-Dihydro-7-[2-(furan-2-yl)ethenyl]-6H-benzopyrano[3,4-c][1,5]benzothiazepine (15). Isolated as white needles in 71% yield, mp 169-170 °C; ¹H-NMR (δ , CDCl₃): 2.73 (1H, m, 6a-H), 4.12 (1H, dd, J = 8.8, 15.7 Hz, 6-H), 4.20 (1H, J = 5.6, 15.7 Hz, 6-H), 4.48 (1H, dd, J = 7.4, 12.1 Hz, 7-H), 6.20 (1H, dd, J = 7.4, 15.1 Hz, α -H), 7.02 (1H, d, J = 15.1 Hz, β -H), 7.20-8.32 (m, 11 arom. H); ¹³C-NMR (δ , CDCl₃): 40.6, 58.5, 66.4, 109.1, 111.4, 117.5, 119.2, 120.0, 121.9, 122.6, 124.8, 125.0, 126.7, 129.9, 133.2, 135.5, 142.2, 151.5, 157.9, 162.6; IR (cm⁻¹): 1602, 1577, 1464, 1377, 1321, 1262, 1212, 1151, 1129, 1014, 958, 927, 814, 774, 752, 687; MS (m/z): 359 (M⁺, 9), 227 (11), 212 (12), 133 (100); Anal. Calcd. for C₂₂H₁₇NO₂S: C, 73.52; H, 4.77; N, 3.89. Found: C, 73.44; H, 4.71; N, 3.92.

6a,7-Dihydro-7-[2-(furan-2-yl)ethenyl]-6H-[1]benzothiopyrano[3,4-c][1,5]benzo- thiazepine (**16**). Isolated as yellow needles in 68% yield, mp 179-180 °C; ¹H-NMR (δ, CDCl₃): 2.72 (1H, m, 6a-H), 3.02 (1H, dd, J = 9.7, 13.8 Hz, 6-H), 3.37 (1H, dd, J = 5.4, 13.8 Hz, 6-H), 4.80 (1H, t, J = 11.4 Hz, 7-H), 6.16 (1H, dd, J = 7.6, 14.9 Hz, α-H), 7.03 (1H, d, J = 14.9 Hz, β-H), 7.09-8.63 (m, 11 arom. H); ¹³C-NMR (δ, CDCl₃): 29.2, 40.5, 58.7, 109.0, 111.4, 118.8, 123.2, 124.7, 125.2, 126.8, 127.6, 128.7, 129.8, 130.9, 135.2, 137.1, 142.1, 144.8, 151.4, 164.3; IR (cm⁻¹): 1601, 1576, 1487, 1462, 1316, 1291, 1257, 1191, 1157, 1013, 957, 928, 884, 784, 756, 735, 593; MS (m/z): 375 (M⁺, 1), 328 (3), 242 (100), 133 (35); Anal. Calcd. for C₂₂H₁₇NOS₂: C, 70.39; H, 4.57, N, 3.73. Found: C, 70.46; H, 4.51; N, 3.67.

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