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ISOMERIZATION IN THE OXIDATIVE CYCLOCONDENSATION OF 2-AROYLMETHYL-1H-BENZIMIDAZOLES WITH *o*-AMINOTHIOPHENOL

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Oxidative cyclocondensation of 2-aroylmethyl-1H-benzimidazoles with o-aminothiophenol gave the previously unknown 3-aryl-2-(2-benzimidazolyl)-4H-1,4-benzothiazines and (or) the isomeric 2H-1,4-benzothiazines. 2-(4-Nitrophenacyl)-1H-benzimidazole and 2-phenacylbenzothiazole gave 4H-1,4-benzothiazines which are not liable to isomerize but the reaction of the first compound is complicated by a hydrolytic fission which yields 2-[2-(4-nitrobenzoylamino)phenylthiomethyl]-benzimidazole. A mixture of dimethylsulfoxide, acetic acid, and water was used as oxidant and solvent. The effect of the substituent and of the solvent on the tendency of the products to undergo prototropic isomerization in the benzothiazine ring have been studied.

Keywords: benzimidazoles, 1,4-benzothiazines, DMSO, isomerization, catalysis, selectivity, cyclocondensation.

We have previously shown that 2-acylmethyl-1H-benzimidazoles are similar in chemical properties to 1,3-dicarbonyl compounds and can serve as efficient synthons in the preparation of benzimidazolyl substituted pyrazoles [1-8], chalcones [9], and pyrimidines [10] and also of (*o*-aminoanilino)pyrazoles [11, 12]. It is also known that 1,3-dicarbonyl compounds undergo oxidative cyclocondensation with o-aminothiophenol (**1**) when heated in DMSO to give 4H-1,4-benzothiazines selectively (e.g. acetoacetic ester gave 2-carbethoxy-3-methyl-4H-1,4-benzothiazine (**2**) [13]). With the aim of preparing previously unavailable benzimidazolyl substituted 1,4-benzothiazines we have used the 2-aroylmethylbenzimidazoles **3a-i** as the methylcarbonyl component in this reaction.

Treatment of reagents **1** and **3a-i** using a known method (holding in DMSO at 145-150°C) led to a mixture of products which was difficult to separate. The individual compounds could be obtained by holding the indicated reagents in mixtures of DMSO–AcOH–H_2O at 85-90°C. It was found that the structure of the products obtained depends on the nature of the aroylmethylbenzimidazole **3** Ar substituent and the composition of the solvent mixture used.

Hence the 2-phenacylbenzimidazole (**3a**) in a mixture of DMSO–AcOH–H_2O (5:1:1 by volume) and its 3-chlorophenacyl analog **3b** in DMSO–AcOH (1:1) gave the target 4H-1,4-benzothiazines **4a,b**. The product **4a** obtained in this way occurred as a crystalline solvate with DMSO (1:1 molecular composition). In a 1:1:1 mixture of DMSO–AcOH–H_2O compounds **3a,b** gave selectively the isomeric 2H-1,4-benzothiazines **5a,b**.

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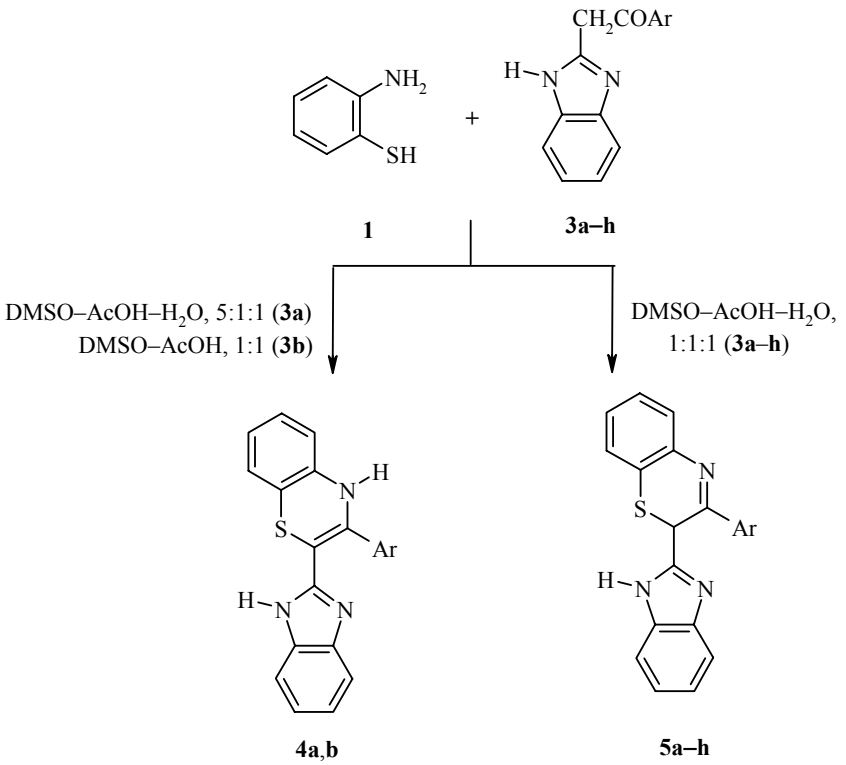
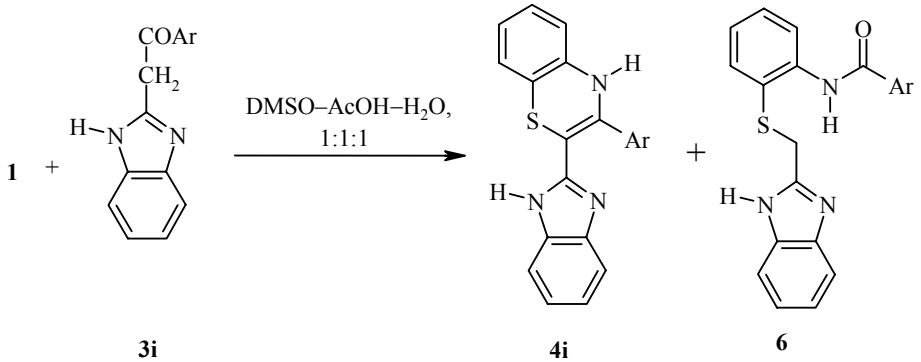


3-5 a Ar = Ph, **b** Ar = 3-ClC_6H_4, **c** Ar = 4-ClC_6H_4, **d** Ar = 4-BrC_6H_4,
e Ar = 3,4,5-$(MeO)_3C_6H_2$, **f** Ar = 4-MeC_6H_4, **g** Ar = 4-$MeOC_6H_4$, **h** Ar = 2-thienyl

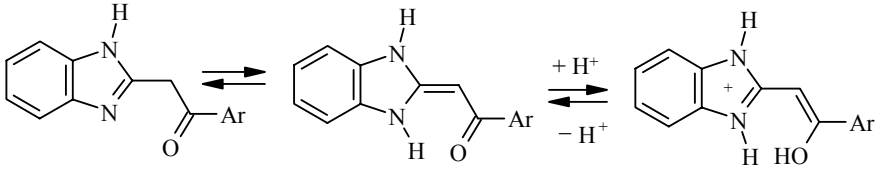
In the case of the 2-aroylmethylbenzimidazoles **3c-h** (compounds with electron-donor or weakly electron-acceptor substituents in the aryl fragment) it was not possible to effect a selective conversion to the corresponding 4H-1,4-benzothiazines **4c-h**. A mixture of DMSO–AcOH–H_2O (1:1:1) gave the isomeric 2H-1,4-benzothiazines **5c-h**.

On the other hand, the same conditions with the 2-(*p*-nitrophenacyl)benzimidazole (**3i**) (a compound with a strong electron acceptor substituent in the aromatic fragment) gave the corresponding 4H-1,4-benzothiazine **4i**. The yield of the latter is only modest (51%) since the process is complicated by the formation of 2-[2-(4-nitrobenzoylamino)phenylthiomethyl]benzimidazole (**6**), evidently due to hydrolytic fission of the target **4i** at the C–C bond of the benzothiazine ring.



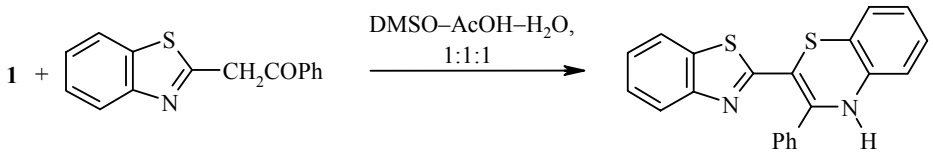
3i, 4i, 6 Ar = 4-$O_2NC_6H_4$

The starting 2-aroylmethylbenzimidazoles **3a-i** principally exist in solution in the enamino ketone form **A** [8, 14], i.e. as benzanilide vinylogs, hence their carbonyl group has a lowered electrophilicity. The protonation of the form **3A** probably occurs at the oxygen atom to give the benzimidazolium derivatives **7a-i**. In structure they resemble the enolized form of acetoacetic ester and, evidently, are more susceptible to reaction with nucleophiles than the nonprotonated forms **A** and **B** and this explains the role of acetic acid as catalyst in the cyclocondensation studied. It does not account for the exclusive formation of the isomers **4** or **5** (see also a detailed mechanism for similar reactions in [13]).



3B a–i **3A a–i** **7a–i**

In order to analyze the obtained results (see below) we have studied the behaviour of a structural analog of compound **3a** in this reaction, *viz* 2-phenacylbenzothiazole **8**. In those conditions for the examples described above which favor the formation of the 2H-isomer product of the product **5** the process takes place markedly more slowly than for compound **3a** (6 h instead of 2 h). None the less, the fully stable 2-(2-benzothiazolyl)-3-phenyl-4H-1,4-benzothiazine (**9**) was obtained in 85% yield.



8 **9**

The physicochemical parameters for the compounds synthesized are given in Table 1.

When considering the reasons for the alternative formation of the isomeric forms of the studied reaction products it should be born in mind that the compounds **4a,b,i, 5a-h** and **9** were obtained by us from the reaction mixture in an analytically pure state by a simple filtration. i.e. without crystallization. In the conditions for recording the ^{1}H NMR spectra (DMSO-d_6, 20-25°C) the appearance of an admixture with the corresponding isomer was not observed. However, attempts to crystallize compound **4b** from aqueous acetic acid or aqueous pyridine showed a tendency to form a mixture of the 2H- and 4H-1,4-benzothiazine isomers as seen in the ^{1}H NMR spectrum. Hence isomeric conversions have a high energetic barrier in this case and, very likely, are catalyzed by acids and water.

In order to understand the tendency of the obtained 1,4-benzothiazines to exist in the 2H- or 4H-isomeric form it seemed reasonable to compare them with compounds **2** and **9**. The latter are stabilized by an energetically favored conjugated system in the 4H-1,4-benzothiazine ring with a transfer of the electron-donor effect of the ring nitrogen atom *via* the vinyl fragment to the electron-acceptor substituent in position 2. Such a system constitutes a chromophoric chain and gives the compounds a red color. A similar conjugation in compounds of type **4** is apparently less energetically favored. This follows from data concerning the weakly developed electron-acceptor properties of a 2-benzimidazolyl fragment [15]. None the less, compound **4i** does not appear prone to isomerization. In this it is possible that another system of conjugation is achieved with transfer of an electron-donor effect from the sulfur atom *via* the vinyl fragment to the strongly electron-acceptor *p*-nitrophenyl substituent. The presence of such a long chromophore is confirmed by the intense dark-cherry color of the compound.

TABLE 1. Characteristics for the Compounds Synthesized

Compound	Empirical formula	Found, % / Calculated, %			mp, °C	Yield, %
		C	H	N		
4a	$C_{21}H_{15}N_3S \cdot C_2H_6OS$	66.11 / 65.84	4.98 / 5.04	10.11 / 10.02	210-215	84
4b	$C_{21}H_{14}ClN_3S$	66.96 / 67.10	3.71 / 3.75	11.12 / 11.18	238-241	63
4i	$C_{21}H_{14}N_4O_2S$	65.35 / 65.27	3.68 / 3.65	14.48 / 14.50	224-226	51
5a	$C_{21}H_{15}N_3S$	73.90 / 73.87	4.37 / 4.43	12.40 / 12.31	238-241	82
5b	$C_{21}H_{14}ClN_3S$	67.04 / 67.10	3.75 / 3.75	11.22 / 11.18	241-243	73
5c	$C_{21}H_{14}ClN_3S$	66.96 / 67.10	3.80 / 3.75	11.09 / 11.18	244-246	88
5d	$C_{21}H_{14}BrN_3S$	60.08 / 60.01	3.33 / 3.36	9.89 / 10.00	258-260	90
5e	$C_{24}H_{21}N_3O_3S$	66.68 / 66.80	5.00 / 4.91	9.76 / 9.74	221-223	52
5f	$C_{22}H_{17}N_3S$	74.45 / 74.34	4.80 / 4.82	11.78 / 11.82	248-250	82
5g	$C_{22}H_{17}N_3OS$	71.08 / 71.14	4.66 / 4.61	11.26 / 11.31	261-263	88
5h	$C_{19}H_{13}N_3S_2$	65.76 / 65.68	3.80 / 3.77	11.98 / 12.09	237-241	89
6	$C_{21}H_{16}N_4O_3S$	62.39 / 62.36	3.94 / 3.99	13.70 / 13.85	194-195	22
9	$C_{21}H_{14}N_2S_2$	70.25 / 70.36	4.02 / 3.94	7.92 / 7.81	220-221	85

The lower stability of type **4** structures with a weakening of the electron-acceptor properties of the Ar substituent is likely attributable to the existence of a concurrent system of conjugation with the oppositely directed transmission of conjugation *via* the vinyl fragment of the electronic effect of thiazine amino group to the benzimidazole fragment. In addition, there can appear a marked steric hindrance between the aryl and benzimidazolyl substituents, positioned to one side of the ring vinyl group. Hence, a deviation of the component parts of the conjugated system from one plane and a lowering of the efficiency of orbital overlap should be expected. As a result, the tendency to prototropic shift with displacement of the multiple bond should be increased and this is found in the structures **5c-h**. In the latter, steric hindrance is not significant. They are evidently additionally stabilized by intramolecular hydrogen bonding between the sulfur atom and the hydrogen atom of the imino group in the benzimidazole ring. A shorter conjugated system for the Ar substituent and the ring azomethine bond is achieved and the chromophoric system is not developed, hence the compounds are colorless or weakly yellowish in color.

As regards the electronic effect on the remaining part of the molecule, the *m*-chlorophenyl substituent in the isomers **4b** and **5b** occupies an intermediate position in the series of Ar substituents used by us between the two indicated limiting examples (see the σ-constant scale [16]) and so both possible isomers can be obtained without particular complications. It seems likely that, in the synthesis using the DMSO–AcOH mixture, protonation of the benzimidazole ring occurs and this increases its electron-acceptor properties to yield the relatively stable conjugated system corresponding to structure **4b**. Treatment of the reaction mixture with water causes rapid hydrolytic decomposition of the salt with the weak acid to give the obtained product. The isomerization of the 1,4-benzothiazine ring occurs significantly more slowly and the product **4b** can be crystallized out in good yield. On the other hand, prolonged heating in the presence of water can result in isomerization to give the product **5b**. The orange color of **4b** and yellow color of its isomer **5b** point to a significant difference in the structure of the chromophoric systems in these compounds.

Somewhat away from the indicated dependence is the example of compound **4a** which is separated as a stable, crystalline solvate with DMSO. The latter is probably formed *via* a stable, intermolecular hydrogen bond between the oxygen atom of the DMSO and the hydrogen atom of the imino group in the benzimidazole ring. There arise specific hindrances to its isomerization in compound **5a** which is stabilized by the intramolecular hydrogen bond. However, when heating is prolonged in a medium containing an increased aqueous acetic acid content the solvate is evidently disrupted and the isomerization can occur without complication. The isomers **4a** and **5a** differ significantly in color, the first being red-orange and the second pale yellow.

The structural features for the synthesized compounds are confirmed by ^{1}H NMR spectroscopic data (Table 2). The signals for the NH groups in compound **4a** (the solvate) and **4b,i** appear as two, single proton singlets to lower field than the aromatic proton signals in a region typical of benzimidazoles [1-12, 14] and 4H-1,4-benzothiazines [13]. Both signals disappear in the presence of D_2O. It was found that, for these compounds, the H-4' and H-7' benzimidazole proton signals appear to be different. In fact, they appear as two resolved multiplets for the solvate **4a** or as a generally broadened singlet signal in the remaining examples. These indicate, respectively, the inhibition and rapid migration of a proton between the ring nitrogen atoms. To

TABLE 2. ^{1}H NMR Spectra of Compounds **4a,b,i, 5a-h, 6,** and **9**

Com-pound	Chemical shifts, δ, ppm (*J*, Hz)
4a DMSO	2.54 (6H, s, $2CH_3$); 6.85-6.95 (3H, m, H-5, H-6, H-7); 7.01-7.09 (3H, m, 8-H, H-5', H-6'); 7.17 (1H, m, H-7'); 7.26-7.40 (5H, m, C_6H_5); 7.46 (1H, m, H-4'); 8.72 (1H, s, H-4); 11.35 (1H, s, H-1')
4b	6.88-6.96 (3H, m, H-5, H-6, H-7); 7.01-7.09 (4H, m, H-8, H-5', H-6', 6-H_{Ar}); 7.30 (1H, dd, J_1 = 8.1, J_2 = 7.8, 5-H_{Ar}); 7.37 (2H, br. s, H-4', H-7'); 7.41-7.46 (2H, m, 2-, 4-H_{Ar}); 8.82 (1H, s, H-4); 11.55 (1H, s, H-1')
4i	6.88-6.96 (3H, m, H-5, H-6, H-7); 7.04-7.11 (3H, m, H-8, H-5', H-6'); 7.34 (2H, br. s, H-4', H-7'); 7.54, 8.17 (2 × 2H, two d, *J* = 8.7, 2-, 6- and 3-, 5-H_{Ar}); 8.91 (1H, s, H-4); 11.69 (1H, s, H-1')
5a	6.14 (1H, s, CH); 7.01-7.54 (9H, m, H-5, H-6, H-7, H-8, H-5', H-6', *m*- and *n*-H_{Ph}); 7.34 (1H, m, H-7'); 7.52 (1H, m, H-4'); 8.13 (2H, m, *o*-H_{Ph}); 12.42 (1H, s, H-1')
5b	6.20 (1H, s, CH); 7.01-7.64 (8H, m, H-5, H-6, H-7, H-8, H-5', H-6', 4,5-H_{Ar}); 7.37 (1H, m, H-7'); 7.54 (1H, m, H-4'); 8.09 (1H, m, 6-H_{Ar}); 8.20 (1H, m, 2-H_{Ar}); 12.46 (1H, s, H-1')
5c	6.15 (1H, s, CH); 7.00-7.42 (7H, m, H-5, H-6, H-7, H-8, H-4', H-6'); 7.51 (1H, m, H-4'); 7.59 and 8.15 (2 × 2H, two d, *J* = 8.4, 2-, 6- and 3-, 5-H_{Ar}); 12.45 (1H, s, H-1')
5d	6.15 (1H, s, CH); 7.01-7.43 (7H, m, H-5, H-6, H-7, H-8, H-4', H-6'); 7.51 (1H, m, H-4'); 7.74 and 8.08 (2 × 2H, two d, *J* = 8.7, 2-, 6- and 3-, 5-H_{Ar}); 12.45 (1H, s, H-1')
5e	3.75 (3H, s, OCP_3); 3.86 (6H, s, 2 OCH_3); 6.27 (1H, s, CH); 7.01-7.42 (6H, m, H-5, H-6, H-7, H-8, H-5', H-6'); 7.35 (1H, m, H-7'); 7.51 (1H, m, H-4'); 7.52 (2H, s, 2-,6-H_{Ar}); 12.35 (1H, s, H-1')
5f	2.37 (3H, s, CH_3); 6.11 (1H, s, CH); 7.01-7.43 (7H, m, H-5, H-6, H-7, H-8, H-4', H-6'); 7.33 and 8.05 (2 × 2H, two d, *J* = 8.7, 2-, 6- and 3-, 5-H_{Ar}); 7.49 (1H, m, H-4'); 12.39 (1H, s, H-1')
5g	3.83 (3H, s, OCH_3); 6.11 (1H, s, CH); 7.01-7.43 (6H, m, H-5, H-6, H-7, H-8, H-5', H-6'); 7.07 and 8.11 (2 × 2H, two d, *J* = 9.0, 2-, 6- and 3-, 5-H_{Ar}); 7.31 (1H, m, H-7'); 7.46 (1H, m, H-4'); 12.37 (1H, s, H-1')
5h	6.15 (1H, s, CH); 7.02-7.40 (7H, m, H-5, H-6, H-7, H-8, H-5', H-6', 4-H_{Ar}); 7.31 (1H, m, H-7'); 7.42 (1H, m, H-4'); 7.85 (2H, m, 3-, 5-H_{Ar}); 12.40 (1H, s, H-1')
6	4.41 (2H, s, CH_2); 7.08-7.11 (2H, m, H-5, H-6); 7.25-7.37 (4H, m, H-3',H-4', H-5', H-6'); 7.31 (1H, m, H-7); 7.74 (1H, m, H-4); 8.26 and 8.39 (2 × 2H, two d, *J* = 8.7, 2-, 6- and 3-, 5-H_{Ar}); 10.84 (1H, s, NHCO); 12.45 (1H, s, H-1)
9	6.74-6.98 (4H, m, H-5, H-6, H-7, H-8); 7.21 (1H, m, H-5'); 7.36 (1H, m, H-6'); 7.48-7.63 (5H, m, C_6H_5); 7.68 (1H, m, H-7'); 7.77 (1H, m, H-4'); 8.96 (1H, s, H-4)

a certain extent the absence of tautomerism in the solvate **4a** is explained by the ring imino group being stably tied to the DMSO by a strong, intermolecular hydrogen bond. In compounds **4b,i** there is no steric hindrance to the corresponding migration which might be due to a deviation of the benzimidazole fragment from the plane of the benzothiazine residue because of steric hindrance on the side of the 3-Ar substituent.

The spectra of the 2H-1,4-benzothiazines **5a-h** are significantly different to those of compound **4**. In these there is noted the presence of a one proton singlet for the CH group which does not disappear in the presence of D_2O and is found to high field of the aromatic protons signals. The singlet signal corresponding to the benzimidazole imino group is shifted by 1 ppm to low field of the analogous signal of the type **4** isomers. Generally, such a shift points to the participation of the imino group of the benzimidazole ring in the formation of an intramolecular hydrogen bond [7, 8, 14] which, in the specific examples, is possible only with the sulfur atom of the benzothiazine ring. Such a hydrogen bond hinders the migration of the proton between the nitrogen atoms of the benzimidazole ring and its H-4' and H-7' protons resonate separately.

The spectrum of compound **6** shows two, one proton singlet signals for the NH groups to low field and these are lost in the presence of D_2O. They correspond to a benzimidazole and an anilide in chemical shift. In the spectrum there are is also present a two proton singlet for the CH_2 group and signals for the aromatic protons whose integrated intensity corresponds to that given in the structure **6**.

The separate appearance of the H-4' and H-7' signals of the benzimidazole fragment point to the formation of an intramolecular hydrogen bond between the proton at position 1 and the sulfur atom of the arylthiomethyl fragment. The structure of the key compounds was also supported by mass spectrometry (Table 3).

Beside data for the molecular weight, essentially the information gives the pattern of molecular fission under electron impact conditions. Hence the spectra of the isomers **4a** and **5a** and also **4b** and **5b** are virtually identical and this supports a ready isomerization in the conditions for acquiring the mass spectra. The spectrum of compound **6** shows signals for the fragments formed by fission of the molecule at the amide C–N bond with values of *m/z* of 150 and 254. A very strong peak for the fragment with mass 131 in it is also characteristic of stable cations. It evidently corresponds to a 2-methylenebenzimidazole cation which is formed as a result of the fission of the molecule at the thiomethylene bond with the capability of delocalizing the charge.

Hence the majority of the discussed 3-aryl-2-(2-benzimidazolyl)-1,4-benzothiazines exist preferentially in the 2H-form due to the weakly expressed electron acceptor properties of the benzimidazole ring and the tendency of its imino group to form an intramolecular bond with the benzothiazine sulfur atom. Stabilization of the 4H-form is a consequence of the strong acceptor substituent in the 3-aryl fragment.

TABLE 3. Mass Spectra of Compounds **4a,b, 5a,b,** and **6**

Com-pound	*m/z* (*I*, %)*
4a	341 $[M]^+$ (100), 308 (29), 238 (84), 223 (58), 170 (22), 78 [M^+, DMSO] (20), 63 (24)
4b	375 $[M]^+$ (59), 257 (49), 245 (21), 238 (100), 170 (20)
5a	341 $[M]^+$ (86), 308 (22), 296 (25), 238 (100), 223 (67)
5b	375 $[M]^+$ (47), 257 (48), 238 (100)
6	404 $[M]^+$ (48), 254 (65), 131 (100), 104 (22), 77 (20)

* Signals with intensities no lower than 20% are given.

EXPERIMENTAL

^{1}H NMR spectra were recorded on a Varian VXR-300 (300 MHz) spectrometer using DMSO-d_6. Mass spectra were taken on an MX 1321 instrument (70 eV, 220°C). Monitoring of the course of the reaction and the purity of the synthesized compounds was performed by the TLC method on Silufol UV-254 plates in the system benzene–ethanol (9:1) and revealed in UV light. The investigated compounds were held in a Fisher drying pistol under water pump vacuum (115°C, 6 h) before carrying out the melting point, elemental analysis, or spectroscopic investigations.

2-(2-Benzimidazolyl)-3-phenyl-4H-1,4-benzothiazine (4a). A mixture of compound **3a** (2 mmol), compound **1** (2.3 mmol), DMSO (14 mmol), and aqueous acetic acid (50%, 0.5 ml) was held for 2 h at 85-90°C. The cooled reaction mixture was filtered and the product **4a** was washed on the filter with 2-propanol.

2-(2-Benzimidazolyl)-3-(3-chlorophenyl)-4H-1,4-benzothiazine (4b). A mixture of compound **3b** (2 mmol), compound **1** (2.3 mmol), DMSO (8 mmol) and glacial acetic acid (1.0 ml) was held for 2 h at 85-90°C. Water (1.0 ml) was added to the cooled reaction mixture and the mixture was warmed with stirring to complete crystallization. The cooled product was filtered and the product **4b** was washed with 2-propanol.

2-(2-Benzimidazolyl)-3-(4-nitrophenyl)-4H-1,4-benzothiazine (4i) and 2-[2-(4-Nitrobenzoylamino)-phenylthiomethyl]benzimidazole (6). A mixture of compound **3i** (2 mmol), compound **1** (2.3 mmol), DMSO (8 mmol), and aqueous acetic acid (50%, 1.0 ml) was heated for 8 h at 85-90°C. Aqueous acetic acid (50%, 1.0 ml) was then added and stirred for 5 min. After cooling, the precipitated product **4i** was filtered off and washed with 2-propanol. The filtrate was held for 8 h at 85-90°C until compound **4i** had disappeared (using TLC). Water (5 ml) was added followed by stirring and cooling in running water. The aqueous layer was poured off, the remaining oil was dissolved in glacial acetic acid (1.5 ml), and concentrated hydrochloric acid (0.5 ml) was added to the solution. Compound **6** hydrochloride fractionally crystallized out at room temperature and it was filtered off and washed with 2-propanol and then converted to the base by the addition to the precipitate of 2-propanol (1.5 ml) and concentrated aqueous ammonia solution (0.5 ml). Crystallization from aqueous pyridine (1:1) gave compound **6**.

2-(2-Benzimidazolyl)-3-phenyl-2H-1,4-benzothiazine (5a). A mixture of compound **3a** (2 mmol), compound **1** (2.3 mmol), DMSO (8 mmol), and aqueous acetic acid (50%, 1.0 ml) was held for 2 h at 85-90°C with stirring for the first 5-10 in until homogeneous. After cooling, the precipitated product **5a** was filtered off and washed with 2-propanol.

In the same way the aroylmethylbenzimidazoles **3b-h** or phenacylbenzothiazole **8** gave compounds **5b-h** or **9** respectively. For the separation of the product **5e** the reaction mixture was diluted with water (2.0 ml). For the preparation of compound **9** the heating was prolonged to 6 h.

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