

STUDY ON REACTION OF 2-ALLYLTHIOBENZIMIDAZOLE WITH BROMINE

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It has been found by ^1H NMR method that bromination of 2-allylthio-benzimidazole proceeds to give the bromocyclization products (benzimidazo-[2,1-*b*]thiazolium and benzimidazo[2,1-*b*][1,3]thiazinium bromides) and two isomeric products of bromine addition to the double bond of allyl moiety.

Keywords: 2-mercaptobenzimidazole, 2-allylthiobenzimidazole, bromocyclization, bromonium and thiiranium ions, ^1H NMR spectroscopy.

Introduction

The chemistry of benzimidazole and particularly 2-mercaptobenzimidazole and its derivatives has received considerable attention because of their synthetic and biological importance. Natural compounds (e.g. vitamin B₁₂) and various medicines (e.g. dibazole, omeprazole, mebendazole, afobazole, etc.) contain benzimidazole moiety in their structure. 2-Mercaptobenzimidazole is widely used in industrial application as a stabilizer and an antioxidant in rubber [1, 2], an adsorbent for some metals [3–5] and a corrosion inhibitor [6]. Besides, many kinds of biological activities have been reported for various N,S-derivatives of 2-mercaptobenzimidazole [7].

The chemistry of fused heterocyclic systems obtained from benzimidazole, as well as synthetic paths to thiazolo[3,2-*a*]benzimidazoles and their chemical properties, has been described in the reviews [8, 9]. However, literature data on electrophilic heterocyclization of S-allyl derivatives of 2-mercaptobenzimidazole (**1**) and 5-ethoxy-2-mercaptobenzimidazole by the treatment with iodine and bromine are limited [10–13]. In the present paper we study the interaction of 2-allylthiobenzimidazole (**2**) with bromine and show unexpected results that are different from earlier data [11].

As demonstrated previously by Korotkikh *et al* [11], the reaction between 2-allylthiobenzimidazole **2** and bromine in acetic acid leads to bromine addition to the double bond and formation of 2-(2,3-dibromopropyl)thiobenzimidazole hydrobromide (**3**) precipitated from the reaction mixture. However, the authors do not explain the origin of HBr in the reaction. The treatment of hydrobromide **3** with mild alkaline agents produces 2-(2,3-dibromopropyl)thiobenzimidazole (**3a**), which undergo cyclization into 3-bromo-2,3,4,10-tetrahydrobenzimidazo[2,1-*b*][1,3]thiazinium bromide (**4**) and isomeric 2*H*-benzimidazo[2,1-*b*][1,3]thiazine (**5**) and 4*H*-benzimidazo[2,1-*b*][1,3]thiazine (**6**) at room temperature in acetone or acetonitrile solution or in the melt (Scheme 1). The mixture of thiazines **5** and **6** is also obtained by refluxing bromide **4** in methanol solution of alkali.

We have earlier reported iodo- and bromocyclization of 3-allylthio-1,2,4-triazoles [14–16]. Notably, bromination of 3-allylthio-1,2,4-triazole and 2-allylthiobenzthiazole with the structure similar to that of compound **2** leads to predominant annelation of thiazole ring. In order to make these issues clear, we have attempted to study the reaction between 2-allylthio-benzimidazole **2** and bromine in more detail.

Experimental

^1H NMR spectra were recorded for DMSO-*d*₆ or CDCl₃ solutions of compounds on a Bruker DRX-400 instrument (400 MHz) using Me₄Si as the internal standard.

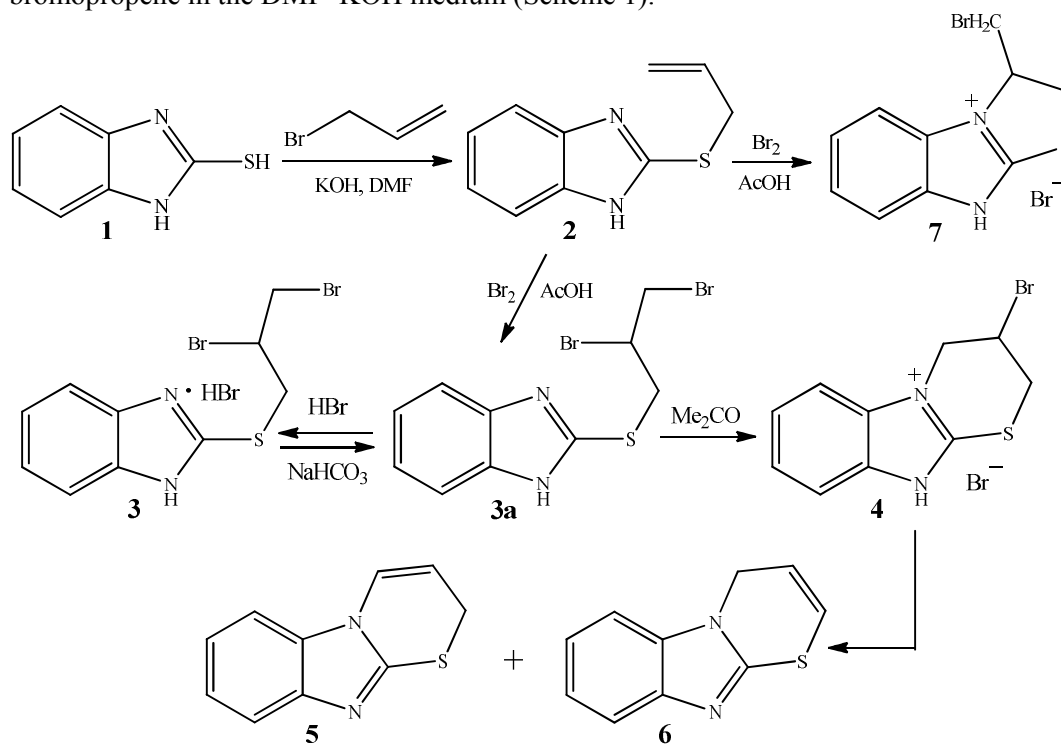
2-Allylthiobenzimidazole (2). To the solution of 2-mercaptobenzimidazole **1** (0.300 g, 2 mmol) in DMF (5 mL) a solution of KOH (0.112 g, 2 mmol) in water (2 mL) and 3-bromopropene (0.18 mL, 2 mmol) were added. After 24 h, the reaction mixture was poured into water (50 mL) and the produced white precipitate was filtered off, washed with water and dried. The precipitate was recrystallized from the hexane–chloroform mixture (1:1) to give compound **2** as a white solid. Yield 0.266 g (70 %), mp 137 °C.

Bromination of 2-allylthiobenzimidazole (2). To a solution of 2-allylthiobenzimidazole **2** (0.190 g, 1 mmol) in acetic acid (3 mL) at 0–5 °C a solution of bromine (0.05 mL, 1 mmol) in acetic

acid (3 mL) was added dropwise. The produced white precipitate was filtered off and washed with acetone. Yield 0.300 g (the mixture of compounds **3**, **4**, **7** and **8**). The solvent was removed from the filtrate; the residue was treated with acetone, after removing acetone a white solid was obtained. Yield 0.103 g (the mixture of compounds **3–8**).

Results and Discussion

Initial 2-allylthiobenzimidazole **2** was synthesized by the reaction of 2-mercapto-benzimidazole **1** with 3-bromopropene in the DMF–KOH medium (Scheme 1).



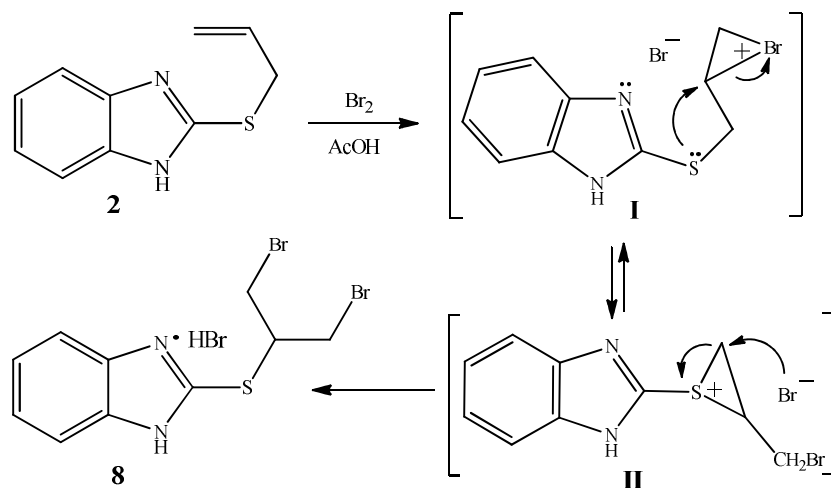
Scheme 1. Synthesis and bromination of 2-allylthiobenzimidazole (**2**)

We have studied the interaction of allyl sulfide **2** with bromine in acetic acid at the allylsulfide **2**:bromine ratio of 1:1.5 under conditions similar to those in [11]. Study of the precipitated product by ^1H NMR method have made it possible to reveal not only hydrobromide **3**, but also benzimidazothiazinium bromide **4** and the second bromocyclization product, 3-bromomethyl-2,3-dihydro-9H-benzimidazo-[2,1-*b*]thiazolium bromide (**7**) resulting from the thiazole ring annelation. From ^1H NMR data, the ratio of compounds **3**, **4** and **7** is $\sim 1.0 : 0.3 : 0.1$. There is a logical question, why only hydrobromide **3** is revealed by the authors of paper [11]. In our opinion, this is due to the fact that ^1H NMR studies were carried out by us on a Bruker instrument (400 MHz), whereas the authors of [11] used a Gemini 200 instrument (200 MHz).

Apparently, bromide **4** was produced *via* intramolecular nucleophilic substitution of adduct **3a**.

The adduct of bromine as hydrobromide **3** was likely formed due to a partial cleavage of HBr from bromides **4** and **7** as a result of their interaction with 2-(2,3-dibromopropyl)thiobenzimidazole **3a**. Thus, bromides **4** and **7** were converted into bases which remained in the solution. This fact may also explain a high prevalence of hydrobromide **3** in the mixture of obtained salts **3**, **4** and **7**.

Notably, a thorough analysis of the ^1H NMR spectra recorded for the resulting mixture has allowed us to identify not only abovementioned compounds **3**, **4** and **7**, but also the adduct of bromine different from compound **3**, namely, 2-[2-bromo-1-(bromomethyl)ethyl]thiobenzimidazole hydrobromide (**8**) (HBr was cleaved from bromides **4** and **7**). It could be produced *via* intermediate formation of bromonium ion (**I**) that was able to undergo intramolecular rearrangement into thiiranium ion (**II**), which was then easily converted into the adduct of bromine of symmetrical structure **8** (Scheme 2). The intramolecular rearrangement of bromonium ion into thiiranium ion has first been studied in the bromination of methylallyl sulfide [17]. In addition, this rearrangement has also been revealed by us during the study of bromination of 3-allylthio-1,2,4-triazoles [16].



Scheme 2. The mechanism of formation of 2-[2-bromo-1-(bromomethyl)ethyl]thiobenzimidazole hydrobromide (8)

^1H NMR spectral data of the synthesized compounds are shown in the table.

 ^1H NMR spectral data of the synthesized compounds

Table

Compound	^1H NMR spectra, δ , ppm (J , Hz)
2	4.04 (2H, d, $^3J = 6.8$, $-\text{SCH}_2-$); 5.07 (1H, m, $=\text{CH}_2$); 5.25 (1H, m, $=\text{CH}_2$); 6.03 (1H, m, $-\text{CH}=\text{}$); 7.11 (2H, m, H_{arom}); 7.46 (2H, m, H_{arom})
3	4.05 (2H, m, $-\text{SCH}_2-$); 4.10 (2H, m, $-\text{CH}_2\text{Br}$); 4.86 (1H, m, $-\text{CHBr}-$); 7.50 (2H, m, H_{arom}); 7.73 (2H, m, H_{arom})
4	3.98 (1H, m, $-\text{SCH}_A\text{H}_B-$); 4.02 (1H, m, $-\text{SCH}_A\text{H}_B-$); 4.61 (1H, m, $-\text{NCH}_M\text{H}_N-$); 4.73 (1H, m, $-\text{NCH}_M\text{H}_N-$); 5.08 (1H, m, $-\text{CH}_X\text{Br}-$); 7.42 (2H, m, H_{arom}); 7.78 (2H, m, H_{arom})
7	4.05 (1H, m, $-\text{CH}_A\text{H}_B\text{Br}$); 4.10 (1H, m, $-\text{CH}_A\text{H}_B\text{Br}$); 4.13 (1H, m, $-\text{SCH}_M\text{H}_N-$); 4.56 (1H, m, $-\text{SCH}_M\text{H}_N-$); 5.19 (1H, m, $-\text{NCH}_X-$); 7.44 (2H, m, H_{arom}); 7.76 (2H, m, H_{arom})
8	3.94 (2H, dd, $^2J = 11.0$, $^3J = 6.6$, $-\text{CH}_2\text{Br}$); 3.98 (2H, dd, $^2J = 11.0$, $^3J = 6.6$, $-\text{CH}_2\text{Br}$); 4.63 (1H, m, $-\text{SCH}<$); 7.52 (2H, m, H_{arom}); 7.73 (2H, m, H_{arom})

A set of proton signals of both bromocyclization products, bromides **4** and **7**, contained signals for aromatic protons shifted downfield and multiplet for the $-\text{CH}_X\text{Br}-$ proton (δ 5.08 ppm) and the $-\text{NCH}_X-$ proton (δ 5.19 ppm), respectively, as well as the characteristic proton signals for such ABMNX spin systems.

In the ^1H NMR spectrum of compound **8** the $-\text{SCH}<$ proton was revealed as multiplet at δ 4.63 ppm. Two-proton doublets of doublets of four $-(\text{CH}_2\text{Br})_2$ protons having a double integral intensity appeared at δ 3.94 and 3.98 ppm.

The filtrate obtained after separation of the mixture of products **3**, **4**, **7**, and **8** has been also studied (such study was not carried out by the authors of paper [11]). After evaporation of acetic acid we managed to obtain a crystalline precipitate which was the mixture of compounds **3**, **4**, **7**, and **8**, as well as 2*H*-thiazine **5** and 4*H*-thiazine **6** (from ^1H NMR data). Formation of thiazines **5** and **6** as by-products of this reaction has also been reported previously [11]. ^1H NMR spectral data produced by us for the mentioned compounds are identical to the published ones [11].

Bromination of compound **2** has also been studied by us in the ^1H NMR experiment. To its solution in CDCl_3 a solution of an equimolar amount of bromine in CDCl_3 was added, and after 1 hour ^1H NMR spectrum of the reaction mixture was recorded. Compound **2** reacted with bromine at once as the spectrum recorded did not contain characteristic signals for the alkenyl protons. From ^1H NMR data, bromine adducts **3** and **8** at the ratio of $\sim 1:3$ predominated in the studied reaction mixture. These results

indicate that the bromocyclization products **4** and **7** were probably formed directly from intermediate bromine adducts **3** and **8**.

To summarize, allyl sulfide **2** reacted with bromine to give the mixture of adducts **3a** and **8**, as well as bromide **7** formed from adduct **8**. Compound **3a** was partially converted to bromide **4**, and partly it reacted with bromide **7** to give hydrobromide **3** via cleavage of HBr from bromide **7**. Thiazines **5** and **6** were formed in minor amounts by the reaction between adduct **3a** or **8** and bromide **4** followed by the removal of two HBr molecules.

Conclusions

It has been found that the interaction of 2-allylthiobenzimidazole **2** with bromine in acetic acid proceeds to give not a single product as reported in [11] but the mixture of two bromocyclization products, 3-bromo-2,3,4,10-tetrahydrobenzimidazo[2,1-*b*][1,3]thiazinium and 3-bromomethyl-2,3-dihydro-9*H*-benzimidazo[2,1-*b*]thiazolium bromides, and two products of bromine addition to the double bond of allyl moiety with symmetrical and asymmetrical structure. It has been found by ¹H NMR experiment that at the initial stage of bromination of 2-allylthiobenzimidazole bromine adducts are formed, and then they are partially converted into the bromocyclization products.

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ИССЛЕДОВАНИЕ РЕАКЦИИ 2-АЛЛИЛТИОБЕНЗИМИДАЗОЛА С БРОМОМ

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Методом ЯМР ^1H установлено, что бромирование 2-аллилтиобензимидазола сопровождается образованием продуктов бромциклизации (бромиды бензимидазо[2,1-*b*]тиазолия и бензимидазо[2,1-*b*][1,3]тиазиния) и двух изомерных друг другу продуктов присоединения брома по двойной связи аллильного фрагмента.

Ключевые слова: 2-меркаптобензимидазол, 2-аллилтиобензимидазол, бромциклизация, бромониевый ион, тираниевый ион, спектроскопия ЯМР ^1H .

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