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The synthesis of Novel 2-Hetarylthiazoles *via* the Stille Reaction

Abstract
A preparative approach to the synthesis of 2-hetaryl thiazoles has been developed *via* the interaction of halothiazoles with stannanes according to the Stille reaction. The most effective catalysts and reaction conditions have been found. It has been determined that the formation of by-products occurs due to specific interaction of the corresponding stannanes with the carbonyl group. The by-products have been isolated and characterized. The mechanism of this interaction with the carbonyl group has not been described in literature. The 2-hetaryl thiazoles obtained have great potential as new building blocks for medicinal chemistry and as ligands due to their complexing properties.
***Keywords*:** Stille reaction; heterocycle; thiazole; Buchwald catalysts; chalcone

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Одержання нових 2-гетарилтіазолів за реакцією Стілле

Анотація
Розроблено препаративний підхід до синтезу 2-гетарилтіазолів, що полягає у взаємодії галогенотіазолів зі станнанами за реакцією Стілле. Визначено найбільш ефективні каталізатори та умови проведення реакції. З'ясовано, що за рахунок специфічної взаємодії відповідних станнанів з карбонільною групою відбувається утворення побічних продуктів, які виділили та схарактеризували. Механізм такої взаємодії з карбонільною групою в літературі не описано. Завдяки своїм комплексоутворювальним властивостям отримані 2-гетарилтіазоли мають великий потенціал як нові будівельні блоки для медичної хімії та як ліганди.
***Ключові слова*:** реакція Стілле; гетероцикл; тіазол; каталізатори Бучвальда; халкон

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■ Introduction

The chemistry of chalcones and their analogs has been actively developed over the past few decades. However, their selective modification remains an important issue [1]. In previous works, chalcones with 2,4-dichlorothiazole moiety were obtained, and ways for their modification with dialkylamino and methoxy groups were shown [2]. Similar dibromo derivatives were also studied (since in several cases their reactivity was much higher), and other important reagents, thiazolylbutenones, were synthesized and modified in the similar synthetic pathway [3]. This fact allowed us to conclude that such thiazole-containing derivatives of diarylideneacetone deserve closer consideration because of their use both in medicine and as ligands for Pd catalysts in chemistry [4–6]. In addition, some other promising thiazole-containing pyrimidines and benzimidazoles were obtained [7] and their versatile properties were studied.

■ Results and discussion

In this work, we propose various approaches for the thiazole ring modification with a series of different substituents implemented. The first of them was thiazole cyclization based on thio-

urea derivatives with its further modification described previously [8]. However, this way did not allow us to reach high diversity of substrates that constituted significant disadvantage. In addition, restrictions on initial thioureas considerably reduce the number of derivatives that can be obtained by this way.

The most successful way for such transformation is the Suzuki and Stille cross-coupling reactions [9, 10]. Our efforts to put 2,4-dihalogenothiazoles to the Suzuki reaction with boronic acids did not show the expected result. On the other hand, they can react with boronic esters, however conditions of the interactions providing sufficient yields have not been defined yet.

Therefore, the Stille reaction was chosen as our main point of interest. The first study was devoted to finding the most suitable catalyst. It is known that $Pd(PPh)_4$ and $Pd_2(dba)_3$ are commonly used in this reactions. There are also known references to the use of the Buchwald precatalysts Pd G3 and Pd G4 with various ligands in this reaction. Moreover, they showed perfect results in a series of other cross-coupling reactions [11], and this fact turned us to assay them in our cases.

Thus, we chose a model reaction of 3-(2,4-dibromothiazolyl)-1-phenyl-propenone with 2-pyridine-tributylstannane to study regioselectivity of the interaction in the presence of a series of catalysts. The reaction was interesting in terms of comparison the reactivity of C2 and C4 positions of 2,4-dibromothiazoles. As a result, the formation of the product mixture was observed in most cases (**Scheme 1**, **Table 1**).

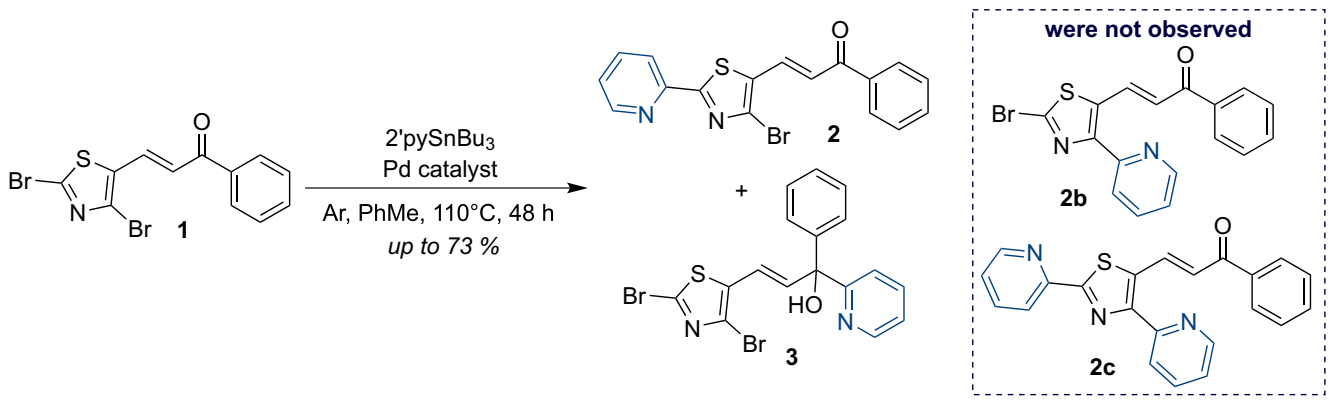
Meanwhile, the assumed products of the reaction in positions C4 (**2b**) or both C2 and C4 (**2c**) were not detected. Nevertheless, we managed to determine the most suitable catalysts for the preparation of both main product **2** (highlighted in green, **Table 1**) and by-product **3** (highlighted in yellow, **Table 1**).

We noted that Pd G3 DavePhos was less suitable for the by-product synthesis than Pd G4 Sphos as evidenced by the *by-product / main product yield ratio*, and with the reaction time variation the second one gave better results. In most cases, a significant number of starting materials remained unreacted in the mixture. In addition, we presumed that catalysts #3 and #4 could be more efficient for compounds without active halogens.

Such a possibility of the carbonyl group in the structure containing reactive bromine atoms to interact first is very promising. In this way, we disclosed a novel reaction type for introducing aryl substituents to carbonyl group of a chalcone fragment. The authors of the ref. [12] reported a similar interaction. However, it was shown only on alkyl derivatives. It should be also noted those chalcones in contrast to the analogs we used did not have an active bromine capable of interacting with tin derivatives. Having obtained such

Table 1. Yields of the products in the Stille reaction of 3-(2,4-dibromothiazolyl)-1-phenyl-propenone with different catalysts (**Scheme 1**)

#	Catalyst	Product **2** yield, %	Product **3** yield, %
1	$Pd(PPh)_4$	40	0
2	Pd G4 XantPhos	0.5	5
3	Pd G4 Sphos	1.3	7.15
4	Pd G3 DavePhos	24.25	14.55
5	Pd G3 CataCXium A	65.2	0
6	Pd G4 *t*-BuXphos	11.1	1.85
7	Pd G3 XantPhos	15.4	7.7
8	Pd G3 AmPhos	73.3	3.8
9	Pd G4 *t*-BuBrettPhos	13.3	3.42
10	$Pd_2(dba)_3$	8.4	4.2
11	Pd G3 SPhos	30.8	5.7
12	Pd G3 Xphos	9.9	1.65
13	Pd G3 *t*-BuBrettPhos	26.4	6.6
14	Pd G4 Xphos	18	10.8
15	Pd G3 *t*-BuXphos	22.2	0



Scheme 1. The Stille reaction of 3-(2,4-dibromothiazolyl)-1-phenyl-propenone with 2-pyridine-tributylstannane

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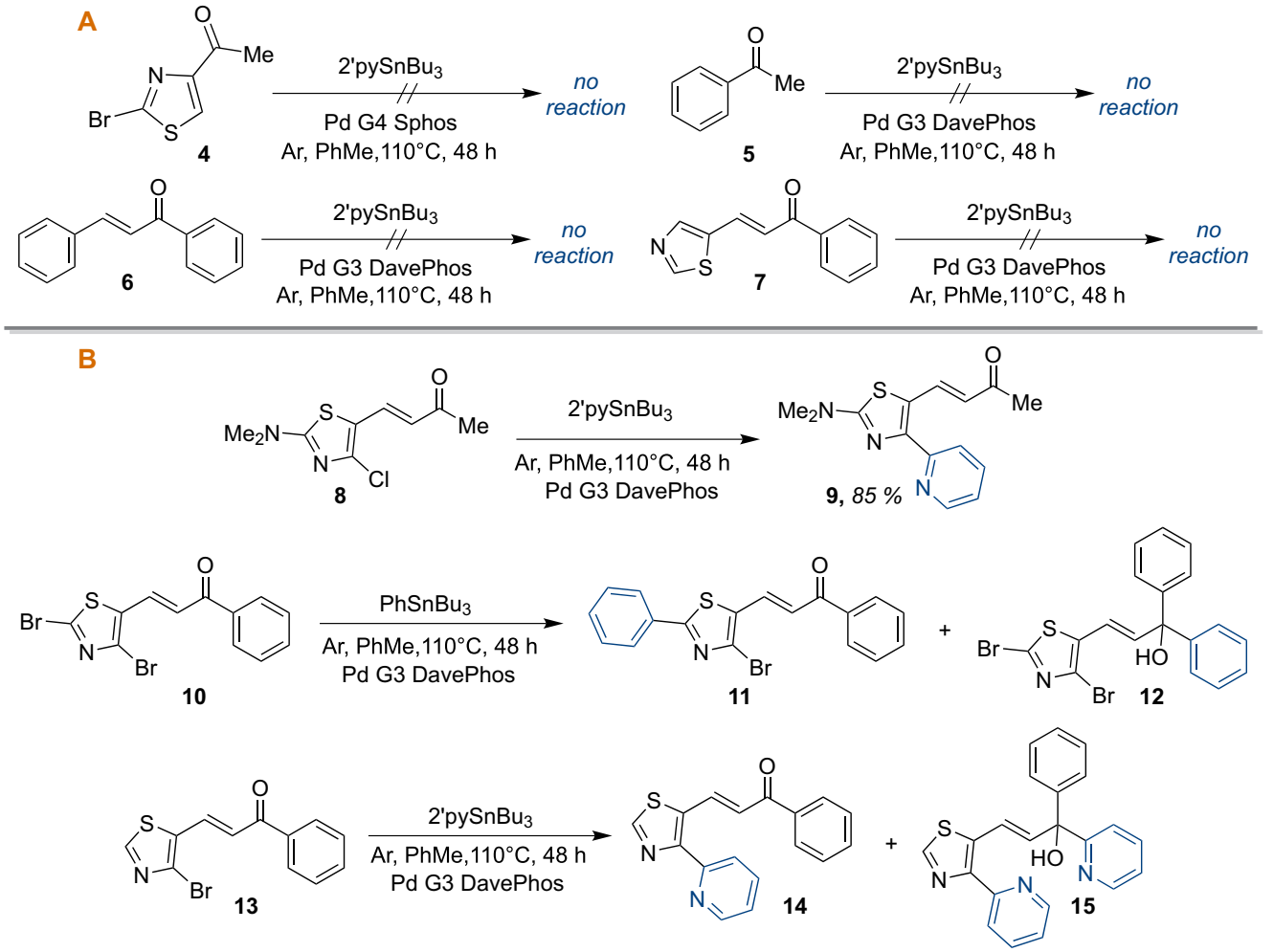


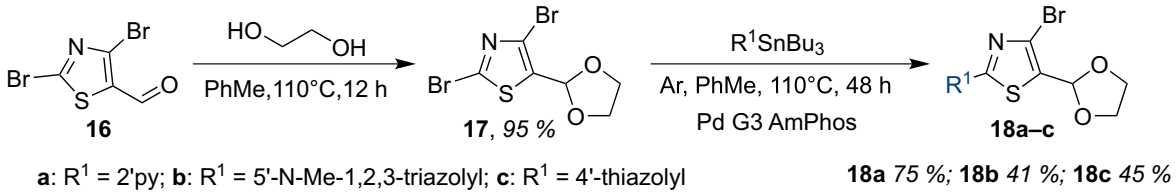
Scheme 2. The study of the reaction scope

an unusual result, we decided to conduct a series of reactions with various carbonyl compounds.

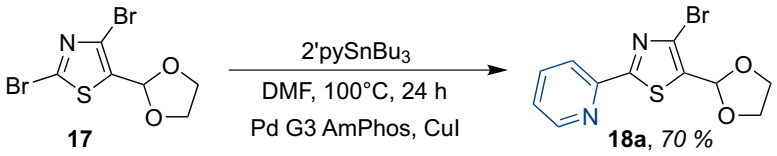
2-Bromo-4-acetylthiazole (**4**) and acetophenone (**5**) were first tested in the reaction. Surprisingly, in the case of Pd G3 DavePhos and Pd G4 Sphos catalysts, we did not observe the product. Taking into account that the conjugated system could lead to another result, chalcone (**6**) and its thiazole analog (**7**) were taken, however, they showed similar result under the same conditions (**Scheme 2**, A). The similar experiment exploiting the same conditions was also carried out for 2'-dimethylamino-4'-chlorothiazol-5'-yl-but-3-en-2-one (**8**). In this case, despite the lower activity of the chlorine as compared to the bromine atom, the reaction proceeded in C4 position giving **9**, and the side product was not observed (**Scheme 2**, B). This fact confirmed the difference in reactivity between 1-methyl and 1-phenyl-3-(thiazol-5-yl)-propenones.

Among other interactions, we studied the reactivity of 4-bromothiazolyl derivative with tributylphenylstannane under the same reaction conditions. The results confirm that 2-pyridile fragment does not play determination role in such interaction. In detail, utilizing 3-(2,4-dibromothiazol-5-yl)-1-phenylprop-2-en-1-one (**10**) under the conditions described above led to two alternative products **11** and **12** (**Scheme 2**, B). In the case of 3-(4-bromothiazol-5-yl)-1-phenylprop-2-en-1-one (**13**), we obtained products of sequential reactions on thiazole ring **14** and carbonyl group **15**. This fact indicates the influence of the bromine atom in position 2 of the ring on the entire thiazolyl-propenone system. Apparently, the results obtained by us are not enough draw convincing conclusions against the background of the observations. Nevertheless, they provide preconditions for further purposeful investigation.

A similar reaction with 2,4-dibromo-5-formylthiazole (**16**) was also carried out, but a mixture of by-products was obtained as a result. For its modification, it was decided to use dioxolane protection of the formyl group. The resulting acetal was introduced into the Stille reaction, which made it possible to diversify a number of products obtained with various stannanes in good yields (**Scheme 3**).



Scheme 3. The pathway to 4-bromo-5-(1,3-dioxolan-2-yl)-2-(hetaryl-2-yl) thiazoles



Scheme 4. Scaling up conditions

Another problem was scaling of the interactions. When 1 g of 3-(2,4-dibromothiazolyl)-1-phenyl-propenone was taken under the same conditions, the yield decreased to 2–7%. To solve this problem, it was proposed to carry out the reaction in DMF with CuI used as a co-catalyst (**Scheme 4**). This method allows conducting such interactions in a bigger scale without the product yields fall.

■ Conclusions

The attractive method of preparative synthesis of the substituted 2-hetaryl thiazoles has been proposed. Buchwald catalysts have shown their effectiveness regardless of the use of the base. The mechanism of the stananne interaction with the carbonyl group is specific and has not been fully studied, which indicates the need to study it. The use of a CuI co-catalyst and the solvent changing to DMF allows scaling of the 2-hetaryl thiazoles synthesis.

■ Experimental part

All chemicals were obtained from Enamine Ltd. and used without further purification. All solvents were purified by standard methods. All procedures were carried out under an open atmosphere with no precautions taken to exclude ambient moisture. ^{1}H NMR spectra were recorded on a Varian MR-400 spectrometer (400 MHz) with TMS as an internal standard. ^{13}C NMR spectra were recorded on a Bruker Avance DRX 500 (126 MHz) spectrometer with TMS as an internal standard. LC-MS spectra were recorded using the chromatography/mass-spectrometric system consisting of a high-performance liquid chromatograph Agilent 1100 LC MSD SL instrument equipped with a diode-matrix and mass-selective detector "Agilent LC/MSD SL". The parameters of chromatography-mass spectrometry analysis were as follows: column – SUPELCO Ascentis Express C18, 2.7 μm 4.6 mm×15 cm. According to the HPLC MS data, all of the compounds synthesized had purity > 95%. The elemental analysis was performed in the Institute of Organic Chemistry of the NASU.

The procedure for the synthesis of 2,4-dibromo-5-(1,3-dioxolan-2-yl)thiazole (17)

2,4-Dibromothiazole-5-carbaldehyde (**16**) (1.0 g, 3.7 mmol) was dissolved in toluene, ethane-1,2-diol (0.69 g, 11.1 mmol), and *p*-toluenesulfonic acid (34 mg, 0.2 mmol) were added. The mixture was refluxed for 24 h and then concentrated under reduced pressure, diluted with 10 mL of water, extracted with *tert*-butyl methyl ether 3×5 mL and washed with brine 3×10 mL. The organic layer was dried over Na_2SO_4, filtered, concentrated under reduced pressure.

A yellow powder. Yield – 1.1 g (95%) Anal. Calcd for $C_6H_5Br_2NO_2S$, %: C 22.88; H 1.60; Br 50.74; N 4.45; O 10.16; S 10.17. Found, %: C 22.87; H 1.60; Br 50.73; N 4.46; S 10.19. ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 3.93–4.12 (4H, m, CH_2-CH_2); 5.96 (1H, s, CH). ^{13}C NMR (126 MHz, $CDCl_3$), δ, ppm: 65.09; 97.84; 122.94; 135.21; 136.66. LC-MS (CI, 200 eV), *m/z* (I_{rel}, %): 314 $[M+H]^+$ (30); 316 (60); 318 (30).

The procedure for the synthesis of 4-bromo-5-(1,3-dioxolan-2-yl)-2-(pyridin-2-yl)thiazole (18a)

Method A. 2,4-Dibromo-5-(1,3-dioxolan-2-yl) thiazole (**17**) (100 mg, 0.32 mmol) and 2-(tributylstannyl)pyridine (117 mg, 0.32 mmol) were placed into a 10 mL flask and dissolved in 4 mL of toluene. Then the mixture was bubbled with argon for 15 min, and a catalyst Pd G3 AmPhos (5 mol %) was added. The reaction mixture was stirred for 48 h at 110°C and cooled to room temperature.

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After that it was separated by flash chromatography (hexane/ethyl acetate, a gradient from 100:0 to 20:80) to give the product.

A yellow powder. Yield – 75 mg (75%). Anal. Calcd for $C_{11}H_9BrN_2O_2S$, %: C 42.19; H 2.90; Br 25.51; N 8.95; O 10.22; S 10.23. Found, %: C 42.18; H 2.90; Br 25.52; N 8.96; S 10.23. ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 3.97–4.23 (4H, m, CH_2-CH_2); 6.14 (1H, s, CH); 7.34 (1H, t, J = 6.2 Hz, N=CH-C<u>H</u>); 7.79 (1H, t, J = 7.8 Hz, N=CH-CH=C<u>H</u>); 8.16 (1H, d, J = 7.8 Hz, N=CH-CH=CH-C<u>H</u>); 8.58 (1H, d, J = 4.8 Hz, N=C<u>H</u>). ^{13}C NMR (126 MHz, $CDCl_3$), δ, ppm: 65.04; 98.51; 119.06; 124.69; 125.08; 133.69; 136.63; 149.01; 149.61; 168.84. LC-MS (CI, 200 eV), m/z (I_{rel}, %): 313 $[M+H]^+$ (100); 315 (98).

Method B. 2,4-Dibromo-5-(1,3-dioxolan-2-yl)-thiazole (**17**) (1.0 g, 3.2 mmol) and 2-(tributylstannyl)pyridine (1.17 g, 3.2 mmol) were placed into a 50 mL flask, dissolved in 20 mL of DMF, then a catalyst Pd G3 AmPhos (5 mol %) and CuI (12 mg, 0.064 mmol) were added. The reaction mixture was stirred for 24 h at 100°C, cooled to room temperature. After that it was separated by flash chromatography (hexane/ethyl acetate, a gradient from 100:0 to 20:80) to give the product. The method provides the product yield of 0.7 g (70%) with all analytical and spectroscopic data being the same to those obtained in the *Method A*.

4-Bromo-5-(1,3-dioxolan-2-yl)-2-(1-methyl-1*H*-1,2,3-triazol-5-yl)thiazole (18b)

The title product was synthesized according to the procedure used for compound **18a** (*Method A*).

A yellow powder. Yield – 41 mg (41%). Anal. Calcd for $C_9H_8BrN_4O_2S$, %: C 34.08; H 2.86; Br 25.19; N 17.67; O 10.09; S 10.11. Found, %: C 34.09; H 2.85; Br 25.17; N 17.68; S 10.12. ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 3.98–4.22 (4H, m, CH_2-CH_2); 4.38 (3H, s, N-CH_3); 6.10 (1H, s, CH); 8.01 (1H, s, C<u>H</u> triazole). ^{13}C NMR (126 MHz, $CDCl_3$), δ, ppm: 13.09; 37.01; 65.19; 98.09; 125.69; 132.42; 133.82; 154.25; 158.54. LC-MS (CI, 200 eV), m/z (I_{rel}, %): 317 $[M+H]^+$ (100); 319 (98).

4-Bromo-5-(1,3-dioxolan-2-yl)-2,4'-bithiazole (18c)

The title product was synthesized according to the procedure used for compound **18a** (*Method A*).

An orange powder. Yield – 46 mg (45%). Anal. Calcd for $C_9H_7BrN_2O_2S_2$, %: C 33.87; H 2.21; Br 25.03; N 8.78; O 10.02; S 20.09. Found, %: C 33.86; H 2.20; Br 25.04; N 8.79; S 20.09. ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 3.86–4.29 (4H, m, CH_2-CH_2); 6.13 (1H, s, CH); 8.08 (1H, s, C=CH-S); 8.82 (1H, s, S-CH-N). ^{13}C NMR (126 MHz, $CDCl_3$), δ, ppm: 65.04; 98.49; 116.47; 125.20; 131.65; 148.74; 153.17; 162.53. LC-MS (CI, 200 eV), m/z (I_{rel}, %): 319 $[M+H]^+$ (100); 321 (98).

The procedure for the synthesis of 3-(2,4-dibromothiazol-5-yl)-1-phenylprop-2-en-1-one (1)

2,4-Dibromothiazole-5-carbaldehyde (**16**) (1.0 g, 3.7 mmol) was dissolved in 10 mL AcOH, then acetophenone (0.44 g, 3.7 mmol) and 0.1 mL of conc. H_2SO_4 were added. The mixture was stirred at 60°C for 24 h and then concentrated under reduced pressure, diluted with 10 mL of water and the precipitate formed was filtered off.

An orange powder. Yield – 0.83 g (60%). Anal. Calcd for $C_{12}H_7Br_2NOS$, %: C 38.64; H 1.89; Br 42.84; N 3.75; O 4.29; S 8.59. Found, %: C 38.63; H 1.88; Br 42.85; N 3.75; S 8.60. ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 7.27 (1H, d, J = 15.5 Hz, C(O)CH); 7.54 (2H, t, J = 7.6 Hz, m-C<u>H</u>(Ph)); 7.63 (1H, t, J = 7.3 Hz, p-C<u>H</u>(Ph)); 7.85 (1H, d, J = 15.4, C(O)CH=C<u>H</u>); 7.99 (2H, d, J = 7.7 Hz, o-C<u>H</u>(Ph)). ^{13}C NMR (126 MHz, $CDCl_3$), δ, ppm: 125.96; 128.50; 128.83; 131.98; 133.42; 134.62; 137.24; 137.60; 188.62. LC-MS (CI, 200 eV), m/z (I_{rel}, %): 372 $[M+H]^+$ (18); 374 (50); 376 (18).

3-(4-Bromo-2-(pyridin-2-yl)thiazol-5-yl)-1-phenylprop-2-en-1-one (2)

The title product was synthesized according to the procedure used for compound **18a** (*Method A*).

An orange powder. Yield – 72 mg (73%). Anal. Calcd for $C_{17}H_{11}BrN_2OS$, %: C 55.00; H 2.99; Br 21.52; N 7.55; O 4.31; S 8.63. Found, %: C 54.98; H 2.99; Br 21.53; N 7.56; S 8.62. ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 7.39 (1H, t, J = 6.2 Hz, N=C-CH=C<u>H</u>); 7.43 (1H, d, J = 15.5, C(O)-C<u>H</u>=CH); 7.51 (2H, t, J = 7.6 Hz, m-C<u>H</u>(Ph)); 7.60 (1H, t, J = 7.3 Hz, p-C<u>H</u>(Ph)); 7.82 (1H, t, J = 7.7 Hz, N=C-C<u>H</u>); 7.93 (1H, d, J = 15.4 Hz, C(O)-CH=C<u>H</u>); 7.99 (2H, d, J = 7.7 Hz, o-C<u>H</u>(Ph)); 8.20 (1H, d, J = 7.9 Hz, N=C-CH=CH-C<u>H</u>); 8.62 (1H, d, J = 4.8 Hz, N-C<u>H</u>). ^{13}C NMR (126 MHz, $CDCl_3$), δ, ppm: 119.72; 124.94; 125.25; 128.03; 128.32; 131.86; 132.59; 132.74; 132.90; 136.82; 137.14; 149.17; 149.30; 168.91; 188.54. LC-MS (CI, 200 eV), m/z (I_{rel}, %): 371 $[M+H]^+$ (100); 373 (90).

3-(4-Bromo-2-(pyridin-2-yl)thiazol-5-yl)-1-phenyl-1-(pyridin-2-yl)prop-2-en-1-ol (3)

The title product was synthesized according to the procedure used for compound **18a** (*Method A*).

An orange powder. Yield – 17 mg (14%). Anal. Calcd for $C_{17}H_{12}Br_2N_2OS$, %: C 45.16; H 2.68; Br 35.34; N 6.20; O 3.54; S 7.08. Found, %: C 45.15; H 2.68; Br 35.32; N 6.21; S 7.09. ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 6.69 (1H, d, J = 15.6 Hz,

C(OH)-CH=CH); 6.88 (1H, d, J = 15.6 Hz, C(OH)-CH=CH); 7.33 (2H, d, J = 7.6 Hz, *o*-CH(Ph)); 7.39 (1H, d, J = 7.7 Hz, N=CH-CH=CH-CH); 7.34 (2H, t, J = 7.5 Hz, *m*-CH(Ph)); 7.37–7.46 (3H, m, N=CH-CH, *p*-CH(Ph)); 8.59 (1H, d, J = 4.8 Hz, N-CH). ^{13}C NMR (126 MHz, $CDCl_3$), δ, ppm: 77.60; 118.82; 121.44; 122.50; 123.19; 126.50; 127.50; 128.08; 133.55; 135.22; 137.26; 139.11; 143.79; 146.92; 160.67. LC-MS (CI, 200 eV), *m*/*z* (I_{rel}, %): 451 $[M+H]^+$ (5); 453 (10); 455 (5); 433 (25); 435 (50); 437 (25).

4-(2-(Dimethylamino)-4-(pyridin-2-yl)-thiazol-5-yl)but-3-en-2-one (9)

The title product was synthesized according to the procedure used for compound **18a** (*Method A*).

An orange powder. Yield – 100 mg (85%). Anal. Calcd for $C_{14}H_{15}N_3OS$, %: C 61.52; H 5.53; N 15.37; O 5.85; S 11.73. Found, %: C 61.51; H 5.52; N 15.36; S 11.75. ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 2.30 (3H, s, C(O)-CH_3); 3.15 (6H, s, CH_3-N-CH_3); 6.10 (1H, d, J = 15.7 Hz, C(O)-CH); 7.20 (1H, d, J = 6.4 Hz, C(O)CH-CH); 7.72 (1H, d, J = 7.6 Hz, N-CH=CH); 8.02 (1H, d, J = 7.9 Hz, N=CH-CH=CH); 8.63 (1H, d, J = 4.9 Hz, N=C-CH); 8.91 (1H, d, J = 15.4 Hz, N-CH). ^{13}C NMR (126 MHz, $CDCl_3$), δ, ppm: 25.99; 39.53; 121.87; 122.28; 123.69; 124.80; 136.14; 137.21; 148.46; 152.90; 153.46; 168.44; 197.84. LC-MS (CI, 200 eV), *m*/*z* (I_{rel}, %): 274 $[M+H]^+$ (100).

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