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SYNTHESIS OF 4-(1*H*-1,2,3-TRIAZOL-4-YL)-1,3-THIAZOLE-2-AMINE DERIVATIVES*

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A series of previously unreported thiazole derivatives, 4-(1H-1,2,3-triazol-4-yl)-1,3-thiazole-2-amines, was prepared by the Hantzsch reaction between the respective 1,2,3-triazole α-bromo ketones and (het)arylthioureas.

Keywords: thioamides, thiazoles, 1,2,3-triazoles, 1,2,3-triazolyl-substituted thiazoles, Hantzsch synthesis, HMBC experiment, HMQC experiment.

Many compounds containing thiazole [1-3] or 1,2,3-triazole [4, 5] fragments exhibit a wide range of biological activity. Therefore it is important to prepare new thiazole derivatives with various polyfunctional substituents, including heterocycles, with the aim of discovering compounds that possess new types of biological activity.

The most frequently used method for the preparation of thiazoles is the Hantzsch synthesis [6], involving a reaction of α-halo-substituted carbonyl compound with compounds containing an N–C–S moiety, such as thioamides [7, 8], and thioureas [9, 10]. This method allows the preparation of polyfunctional thiazoles with diverse substituents.

In the current work, we studied the possibility of using Hantzsch reaction for the preparation of new polyfunctional thiazoles **3a-i** containing a 1,2,3-triazole fragment as one of the substituents. For this purpose, 4-acetyl-1,2,3-triazoles **1a-c** were converted to the respective α-bromo ketones **2a-c** by treatment with elemental bromine in AcOH at 80°C. Their subsequent regioselective cyclocondensation with *N*-(pyridyl)arylthioureas in refluxing ethanol (7 h) led to the formation of 4-(1*H*-1,2,3-triazol-4-yl)-1,3-thiazole-2-amine derivatives **3a-i** in good yields (69-95%).

It should be noted that, despite the presence of an electron-withdrawing 1,2,3-triazole substituent in the molecules of α-bromocarbonyl compounds **2a-c**, the target triazoles were obtained in moderate to high yields. Thus, in the case of compounds **2a**,**b**, the highest yields were obtained in reactions with *N*-arylthioureas containing an electron-donating methyl group at the *para* and *meta* positions, while the lowest yields were obtained in the reaction with *N*-(pyridin-2-yl)thiourea. In the case of α-bromo ketone **2c**, the reaction yields did not depend on substituents in the starting thioureas.

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*Dedicated to Academician O. N. Chupakhin on the occasion of his 80th birthday.

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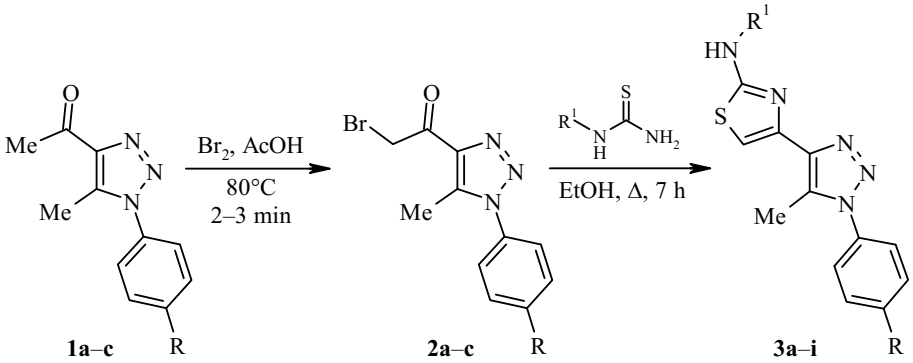

1a, **2a**, **3a**–**c** R = Cl, **1b**, **2b**, **3d**–**f** R = Br, **1c**, **2c**, **3g**–**i** R = NO_2;
3 a,**d**,**g** R^1 = 4-MeC_6H_4, **b**,**e**,**h** R^1 = 3-MeC_6H_4, **c**,**f**,**i** R^1 = 2-Py

The structure of the obtained compounds was unequivocally established by using ^1H and ^{13}C NMR spectroscopy. The ^1H NMR spectra of compounds **3a-i** contained a singlet at 7.25-7.42 ppm due to the thiazole ring proton H-5, besides the characteristic signals of the protons of aryl substituent in the 1,2,3-triazole fragment and those of the arylthiourea fragment. The ^{13}C NMR spectra contained signals due to the C-2,4,5 carbon atoms of the thiazole fragment at 160.3-164.4, 141.5-143.0, and 104.0-107.9 ppm, respectively. Complete assignment of ^{13}C NMR signals of the synthesized compounds was based on ^1H–^{13}C HMQC and ^1H–^{13}C HMBC techniques (Fig. 1).

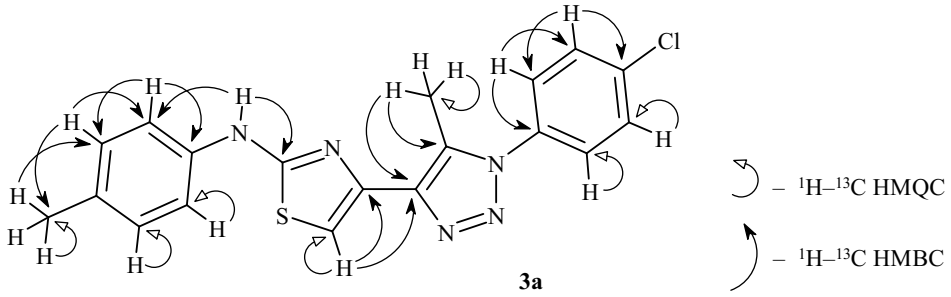

Fig. 1. The ^1H–^{13}C heteronuclear correlations for compound **3a** according to ^1H–^{13}C HMQC and ^1H–^{13}C HMBC experiments.

The ^1H–^{13}C HMQC spectra of compound **3a** contained all ^1H–^{13}C correlations through one bond, which allowed to assign the ^{13}C NMR signals of carbon atoms bonded to hydrogen. The presence of ^1H–^{13}C correlation cross peaks through two and three bonds in the ^1H–^{13}C HMBC spectra allowed to unequivocally assign the quaternary ^{13}C NMR signals of aryl substituents, thiazole and 1,2,3-triazole rings.

Thus, the Hantzsch reaction of (bromomethyl)(1,2,3-triazol-4-yl)ketones with (het)arylthiourea gave a series of previously unreported thiazole derivatives – 4-(1*H*-1,2,3-triazol-4-yl)-1,3-thiazole-2-amines.

EXPERIMENTAL

IR spectra were recorded on an FSM-1201 instrument in KBr pellets. ^1H and ^{13}C NMR spectra were acquired on a Bruker Avance spectrometer (600 and 150 MHz, respectively) in DMSO-d_6, internal standard was TMS. The ^1H–^{13}C HMBC and ^1H–^{13}C HMQC experiments were performed on the same spectrometer. Elemental analysis was performed on a vario EL cube analyzer. Melting points were determined on a Boetius hot stage and were not corrected.

The starting ketones **1a-c** were obtained according to a published method [11], substituted thioureas – according to another known method [12].

Synthesis of Compounds 2a-c (General Method). A solution of ketone **1a-c** (10 mmol) in AcOH (20 ml) was heated to 80°C and treated by several episodes of dropwise addition of Br_2 (0.51 ml, 1.6 g, 10 mmol) in AcOH (5 ml). After the color faded (2-3 min), the solution was cooled and treated with H_2O (10 ml). The obtained precipitate was filtered off and recrystallized from EtOH.

2-Bromo-1-[1-(4-chlorophenyl)-5-methyl-1*H*-1,2,3-triazol-4-yl]ethanone (2a). Yield 84%, white crystals, mp 118-119°C. IR spectrum, ν, cm^{-1}: 3298, 1696, 1548, 1497, 1178, 962, 877. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.55 (3H, s, CH_3); 4.88 (2H, s, CH_2Br); 7.72 (2H, d, *J* = 8.7, H Ar); 7.75 (2H, d, *J* = 8.7, H Ar). Found, %: C 42.07; H 2.92. $C_{11}H_9BrClN_3O$. Calculated, %: C 42.00; H 2.88.

2-Bromo-1-[1-(4-bromophenyl)-5-methyl-1*H*-1,2,3-triazol-4-yl]ethanone (2b). Yield 83%, white crystals, mp 125-126°C. IR spectrum, ν, cm^{-1}: 3301, 1693, 1550, 1495, 1181, 972, 872. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.55 (3H, s, CH_3); 4.88 (2H, s, CH_2Br); 7.62 (2H, d, *J* = 8.8, H Ar); 7.87 (2H, d, *J* = 8.8, H Ar). Found, %: C 36.85; H 2.61. $C_{11}H_9Br_2N_3O$. Calculated, %: C 36.80; H 2.53.

2-Bromo-1-[5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl]ethanone (2c). Yield 76%, white crystals, mp 135-136°C. IR spectrum, ν, cm^{-1}: 3305, 1672, 1604, 1532, 1313, 963, 823. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.63 (3H, s, CH_3); 4.90 (2H, s, CH_2Br); 8.01 (2H, d, *J* = 8.8, H Ar); 8.50 (2H, d, *J* = 8.8, H Ar). Found, %: C 40.70; H 2.83. $C_{11}H_9BrN_4O_3$. Calculated, %: C 40.64; H 2.79.

Synthesis of Compounds 3a-i (General Method). A mixture of α-bromo ketone **2a-c** (1.5 mmol) and the respective thiourea (1.5 mmol) was refluxed in EtOH (30 ml) for 7 h. Then the solution was treated with saturated $NaHCO_3$ solution (10 ml). The mixture was cooled, and the obtained precipitate was filtered off and recrystallized from EtOH.

4-[1-(4-Chlorophenyl)-5-methyl-1*H*-1,2,3-triazol-4-yl]-*N*-(4-methylphenyl)-1,3-thiazole-2-amine (3a). Yield 81%, yellow crystals, mp 203-205°C. IR spectrum, ν, cm^{-1}: 3364, 1612, 1536, 1497, 1316, 1089, 1008, 803. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.25 (3H, s, ArC$\underline{H}_3$); 2.66 (3H, s, HetC$\underline{H}_3$); 7.12 (2H, d, *J* = 7.9, H Ar); 7.25 (1H, s, H-5 thiazole); 7.57 (2H, d, *J* = 7.9, H Ar); 7.71-7.73 (4H, m, H Ar); 10.22 (1H, s, NH). ^{13}C NMR spectrum, δ, ppm: 10.1 (Het$\underline{C}H_3$); 20.8 (Ar$\underline{C}H_3$); 104.0 (C-5 thiazole); 117.5 (CH Ar); 127.5 (CH Ar); 129.8 (CH Ar); 130.1 (CH Ar); 130.7 (C Ar); 131.1 (C Ar); 134.6 (C triazole); 135.2 (C Ar); 139.2 (C Ar); 140.6 (C triazole); 143.0 (C-4 thiazole); 164.4 (C-2 thiazole). Found, %: C 59.85; H 4.29; N 18.42. $C_{19}H_{16}ClN_5S$. Calculated, %: C 59.76; H 4.22; N 18.34.

4-[1-(4-Chlorophenyl)-5-methyl-1*H*-1,2,3-triazol-4-yl]-*N*-(3-methylphenyl)-1,3-thiazole-2-amine (3b). Yield 95%, yellow crystals, mp 198-199°C. IR spectrum, ν, cm^{-1}: 3352, 1609, 1543, 1525, 1498, 1093, 1008, 837. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.29 (3H, s, ArC$\underline{H}_3$); 2.70 (3H, s, HetC$\underline{H}_3$); 6.78 (1H, d, *J* = 7.5, H Ar); 7.19 (1H, t, *J* = 7.8, H Ar); 7.28 (1H, s, H-5 thiazole); 7.37 (1H, d, *J* = 8.1, H Ar); 7.69 (1H, s, H Ar); 7.70-7.74 (4H, m, H Ar); 10.26 (1H, s, NH). ^{13}C NMR spectrum, δ, ppm: 10.1 (Het$\underline{C}H_3$); 21.8 (Ar$\underline{C}H_3$); 104.2 (C-5 thiazole); 114.6 (CH Ar); 118.0 (CH Ar); 122.5 (CH Ar); 127.5 (CH Ar); 129.3 (CH Ar); 130.1 (CH Ar); 131.1 (C Ar); 134.6 (C triazole); 135.2 (C Ar); 138.6 (C Ar); 140.6 (C Ar); 141.5 (C triazole); 143.0 (C-4 thiazole); 164.2 (C-2 thiazole). Found, %: C 59.87; H 4.26; N 18.39. $C_{19}H_{16}ClN_5S$. Calculated, %: C 59.76; H 4.22; N 18.34.

***N*-{4-[1-(4-Chlorophenyl)-5-methyl-1*H*-1,2,3-triazol-4-yl]-1,3-thiazol-2-yl}pyridine-2-amine (3c)**. Yield 69%, light-yellow crystals, mp 233-234°C. IR spectrum, ν, cm^{-1}: 3243, 1607, 1541, 1496, 1480, 1412, 1302, 1090, 831, 772. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.67 (3H, s, CH_3); 6.95 (1H, t, *J* = 6.9, H-3 Py); 7.13 (1H, d, *J* = 8.3, H-5 Py); 7.38 (1H, s, H-5 thiazole); 7.70-7.75 (5H, m, H Ar, H-4 Py); 8.33 (1H, d, *J* = 4.9, H-6 Py); 11.39 (1H, s, NH). ^{13}C NMR spectrum, δ, ppm: 10.2 (CH_3); 107.4 (C-5 thiazole); 111.3 (CH Py); 116.5 (CH Py); 127.4 (CH Ar); 130.1 (CH Ar); 131.1 (C Ar); 134.6 (C triazole); 135.3 (C Ar); 138.4 (C-4 Py); 140.7 (C triazole); 141.9 (C-4 thiazole); 146.9 (C-6 Py); 152.2 (C-2 Py); 160.3 (C-2 thiazole). Found, %: C 55.41; H 3.58; N 22.86. $C_{17}H_{13}ClN_6S$. Calculated, %: C 55.36; H 3.55; N 22.78.

4-[1-(4-Bromophenyl)-5-methyl-1*H*-1,2,3-triazol-4-yl]-*N*-(4-methylphenyl)-1,3-thiazole-2-amine (3d). Yield 76%, cream-colored crystals, mp 209-210°C. IR spectrum, ν, cm^{-1}: 3360, 1611, 1536, 1443, 1317, 1239, 1065, 1006, 830, 503. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.25 (3H, s, ArC$\underline{H}_3$); 2.66 (3H, s, HetC$\underline{H}_3$); 7.12 (2H, d, *J* = 6.9, H Ar); 7.25 (1H, s, H-5 thiazole); 7.58 (2H, d, *J* = 7.3, H Ar); 7.65 (2H, d, *J* = 7.3, H Ar); 7.84 (2H, d, *J* = 7.3, H Ar); 10.24 (1H, s, NH). ^{13}C NMR spectrum, δ, ppm: 10.1 (Het$\underline{C}H_3$); 20.8 (Ar$\underline{C}H_3$); 104.0 (C-5 thiazole); 117.5 (CH Ar); 123.2 (C Ar); 127.7 (CH Ar); 129.8 (CH Ar); 130.6 (C Ar); 131.1 (C Ar); 133.1 (CH Ar); 135.6 (C triazole); 139.2 (C Ar); 140.6 (C triazole); 143.0 (C-4 thiazole); 164.4 (C-2 thiazole). Found, %: C 53.61; H 3.83; N 16.51. $C_{19}H_{16}BrN_5S$. Calculated, %: C 53.53; H 3.78; N 16.43.

4-[1-(4-Bromophenyl)-5-methyl-1*H*-1,2,3-triazol-4-yl]-*N*-(3-methylphenyl)-1,3-thiazole-2-amine (3e). Yield 78%, cream-colored crystals, mp 211-212°C. IR spectrum, ν, cm^{-1}: 3356, 1608, 1542, 1525, 1495, 1309, 1070, 1005, 835, 552. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.29 (3H, s, ArC$\underline{H}_3$); 2.70 (3H, s, HetC$\underline{H}_3$); 6.78 (1H, d, *J* = 7.3, H Ar); 7.19 (1H, t, *J* = 7.8, H Ar); 7.28 (1H, s, H-5 thiazole); 7.37 (1H, d, *J* = 8.2, H Ar); 7.66 (2H, d, *J* = 8.2, H Ar); 7.70 (1H, s, H Ar); 7.82-7.89 (2H, m, H Ar); 10.28 (1H, s, NH). ^{13}C NMR spectrum, δ, ppm: 10.1 (Het$\underline{C}H_3$); 21.8 (Ar$\underline{C}H_3$); 104.2 (C-5 thiazole); 114.6 (CH Ar); 117.9 (CH Ar); 122.5 (CH Ar); 127.7 (CH Ar); 129.2 (CH Ar); 131.1 (C Ar); 133.1 (CH Ar); 133.2 (C triazole); 135.6 (C Ar); 138.6 (C Ar); 140.6 (C triazole); 141.5 (C Ar); 142.9 (C-4 thiazole); 164.2 (C-2 thiazole). Found, %: C 53.59; H 3.82; N 16.48. $C_{19}H_{16}BrN_5S$. Calculated, %: C 53.53; H 3.78; N 16.43.

***N*-{4-[1-(4-Bromophenyl)-5-methyl-1*H*-1,2,3-triazol-4-yl]-1,3-thiazole-2-yl}pyridine-2-amine (3f)**. Yield 79%, white crystals, mp 245-246°C. IR spectrum, ν, cm^{-1}: 3323, 1608, 1536, 1496, 1415, 1299, 1069, 1005, 832, 771. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.67 (3H, s, CH_3); 7.12 (1H, d, *J* = 8.3, H-3 Py); 7.37 (1H, s, H-5 thiazole); 7.62 (1H, d, *J* = 8.6, H-5 Py); 7.65-7.67 (2H, m, H Ar); 7.70-7.75 (1H, m, H-4 Py); 7.86 (2H, d, *J* = 8.7, H Ar); 8.33 (1H, d, *J* = 5.0, H-6 Py); 11.38 (1H, s, NH). ^{13}C NMR spectrum, δ, ppm: 10.2 (CH_3); 107.4 (C-5 thiazole); 111.3 (CH Py); 116.5 (CH Py); 127.7 (CH Ar); 130.1 (CH Ar); 131.1 (C Ar); 134.7 (C triazole); 135.7 (C Ar); 138.4 (CH Py); 140.7 (C triazole); 141.8 (C-4 thiazole); 146.9 (CH Py); 152.2 (C-2 Py); 160.3 (C-2 thiazole). Found, %: C 49.46; H 3.21; N 20.41. $C_{17}H_{13}BrN_6S$. Calculated, %: C 49.40; H 3.17; N 20.33.

4-[5-Methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl]-*N*-(4-methylphenyl)-1,3-thiazole-2-amine (3g). Yield 85%, yellow crystals, mp 183-184°C. IR spectrum, ν, cm^{-1}: 3371, 1614, 1597, 1545, 1526, 1346, 1119, 858. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.25 (3H, s, ArC$\underline{H}_3$); 2.75 (3H, s, HetC$\underline{H}_3$); 7.13 (2H, d, *J* = 8.1, H Ar); 7.29 (1H, s, H-5 thiazole); 7.57 (2H, d, *J* = 8.3, H Ar); 8.02 (2H, d, *J* = 8.9, H Ar); 8.48 (2H, d, *J* = 8.9, H Ar); 10.25 (1H, s, NH). ^{13}C NMR spectrum, δ, ppm: 10.3 (Het$\underline{C}H_3$); 20.8 (Ar$\underline{C}H_3$); 104.4 (C-5 thiazole); 117.5 (CH Ar); 125.6 (CH Ar); 126.4 (CH Ar); 129.8 (CH Ar); 130.7 (C Ar); 131.3 (C triazole); 139.1 (C Ar); 141.0 (C Ar); 141.2 (C triazole); 142.7 (C-4 thiazole); 147.9 (C Ar); 164.5 (C-2 thiazole). Found, %: C 58.22; H 4.17; N 21.48. $C_{19}H_{16}N_6O_2S$. Calculated, %: C 58.15; H 4.11; N 21.41.

4-[5-Methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl]-*N*-(3-methylphenyl)-1,3-thiazole-2-amine (3h). Yield 93%, yellow crystals, mp 227-229°C. IR spectrum, ν, cm^{-1}: 3374, 1613, 1595, 1549, 1527, 1343, 1189, 1088, 1004, 857, 774, 688. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.30 (3H, s, ArC$\underline{H}_3$); 2.79 (3H, s, HetC$\underline{H}_3$); 6.78 (1H, d, *J* = 7.2, H Ar); 7.19 (1H, t, *J* = 7.7, H Ar); 7.32 (1H, s, H-5 thiazole); 7.37 (1H, d, *J* = 7.6, H Ar); 7.70 (1H, s, H Ar); 8.02 (2H, d, *J* = 8.9, H Ar); 8.48 (2H, d, *J* = 8.9, H Ar); 10.30 (1H, s, NH). ^{13}C NMR spectrum, δ, ppm: 10.3 (Het$\underline{C}H_3$); 21.8 (Ar$\underline{C}H_3$); 104.6 (C-5 thiazole); 114.6 (CH Ar); 118.0 (CH Ar); 122.5 (CH Ar); 125.5 (CH Ar); 126.4 (CH Ar); 129.2 (CH Ar); 131.3 (C triazole); 138.6 (C Ar); 141.0 (C Ar); 141.2 (C triazole); 141.5 (C Ar); 142.6 (C-4 thiazole); 147.9 (C Ar); 164.3 (C-2 thiazole); Found, %: C 58.19; H 4.16; N 21.51. $C_{19}H_{16}N_6O_2S$. Calculated, %: C 58.15; H 4.11; N 21.41.

***N*-{4-[5-Methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl]-1,3-thiazol-2-yl}pyridine-2-amine (3i)**. Yield 92%, yellow crystals, mp >300°C (decomp.). IR spectrum, ν, cm^{-1}: 3389, 1607, 1534, 1502, 1481, 1344, 1149, 921, 858. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.76 (3H, s, CH_3); 6.95 (1H, t, *J* = 6.2, H-3 Py); 7.13 (1H, d, *J* = 8.3, H-5 Py); 7.42 (1H, s, H-5 thiazole); 7.71-7.76 (1H, m, H-4 Py); 8.03 (2H, d, *J* = 8.9, H Ar); 8.33 (1H, d, *J* = 4.9, H Py); 8.48 (2H, d, *J* = 8.8, H Ar); 11.42 (1H, s, NH). ^{13}C NMR spectrum, δ, ppm: 10.4 (CH_3); 107.9 (C-5 thiazole); 111.4 (CH Py); 116.7 (CH Py); 125.6 (CH Ar); 126.4 (CH Ar); 131.3 (C triazole); 138.5

(CH Py); 141.2 (C Ar); 141.3 (C triazole); 141.5 (C-4 thiazole); 146.8 (CH Py); 147.9 (C Ar); 152.1 (C-2 Py); 160.4 (C-2 thiazole). Found, %: C 53.88; H 3.49; N 25.92. $C_{17}H_{13}N_7O_2S$. Calculated, %: C 53.82; H 3.45; N 25.84.

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