One-Pot Synthesis of Iimidazo[1,2-*c*]quinazoline-5(6*H*)-thione and Imidazo[1,2-*c*]quinazolin-5(6*H*)-one with the Aid of Tin(II) Chloride

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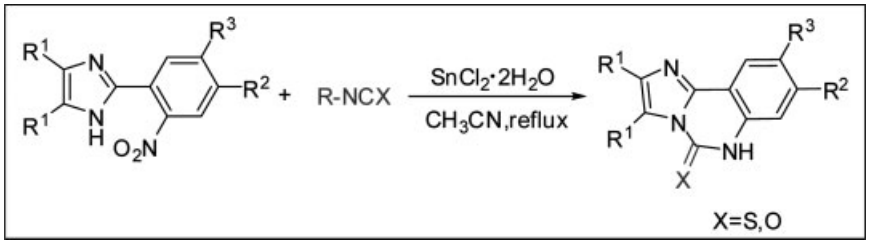

An efficient, convenient, one-pot synthesis of imidazo[1,2-*c*]quinazoline-5(6*H*)-thione and imidazo[1,2-*c*]quinazolin-5(6*H*)-one was accomplished in good yields via the novel reductive cyclization of 2-(2-nitrophenyl)-1*H*-imidazoles with isothiocyanates or isocyanates mediated by $SnCl_2 \cdot 2H_2O$ system.

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INTRODUCTION

A literature survey revealed that quinazolinones show antihypertonic, antirheumatic, antianaphylactic, antiasthmatic, tranquilizing, neurostimulating, and benzodiazepine binding activity [1,2]. For example, 3-substituted quinazolinones, such as SGB-1534 (**1**) [3] and ketanserin (**2**) have been found to have antihypertensive activities mediated via α-adrenoceptor and serotonic receptor anatgonism, respectively. Addition of a (2-methoxyphenyl)piperazine side chain at the 2- or 3-position of the angular tricyclic 2,3-dihydroimidazo[1,2-*c*]quinazoline ring system of SGB-1534 resulted in the formation of potent antihypertensive agents, such as 2-[[4-(2-methoxyphenyl)- piperazin-1-yl]methyl]-2,3-dihydroimidazo[1,2-*c*]-quinazolin-5(6*H*)-one (**3**) and 3-[[4-(2-methoxy phenyl)piperazin-1-yl]methyl]-2,3-dihydroimidazo[1,2-*c*]-quinazolin-5(6*H*)-one (**4**) that selectively antagonized the α_1-adrenoceptor [4]. (Fig. 1)

Imidazo[1,2-*c*]quinazolinone derivatives also are of considerable interest because of their biological and pharmacological activities, including antimicrobial, antihypertension, diuresis, and muscle relaxation [5,6].

The imidazo[1,2-*c*]quinazolinone ring system can generally be prepared by the reaction of α-aminoketones with 2-isocyanatobenzonitrile [7], α-aminocarboxylic esters with 2-isocyanatobenzonitrile [8], and 2-(2-nitrophenyl)-1*H*-imidazoles with triphosgene [9]. Chern et al. also have reported the synthesis of imidazo[1,2-*c*]quinazolinone derivatives based on cyclocondensations of NBS [10]. However, these methods suffer from some disadvantages, such as drastic conditions, unsatisfactory yields, long reaction time, high temperature, complex manipulation, and inaccessible starting materials. Therefore, we became interested in developing a convenient synthetic methods for the preparation of imidazo[1,2-*c*]quinazolinone derivatives.

In recent years, our interest has been focused on the usage of $SnCl_2$ reagent. We have previously reported the synthesis of 2-aryl-2*H*-indazoles [11], 1-hydroxy quinazolinones [12], respectively mediated by $SnCl_2$ reagent. As our earlier works goes, herein, we will describe a new approach to synthesize imidazo[1,2-*c*]quinazolinone derivatives by treating 2-(2-nitrophenyl)-1*H*-imidazoles with isothiocyanates or isocyanates mediated by $SnCl_2$ reagent.

RESULTS AND DISCUSSION

On the basis of our previous experience, we selected 2-(2-nitrophenyl)-4,5-diphenyl-1*H*-imidazole **1a** and the 4-methylphenyl isothiocyanate **2a** as model substrates to optimize the experimental conditions for the proposed reductive cyclization reaction (Scheme 1). The results are summarized in Table 1.

As shown in Table 1, we briefly examined the effect of different temperatures, different solvents and ratio of **1a**:$SnCl_2 \cdot 2H_2O$. The results showed that at reflux the reaction proceeded smoothly in high yield. To further

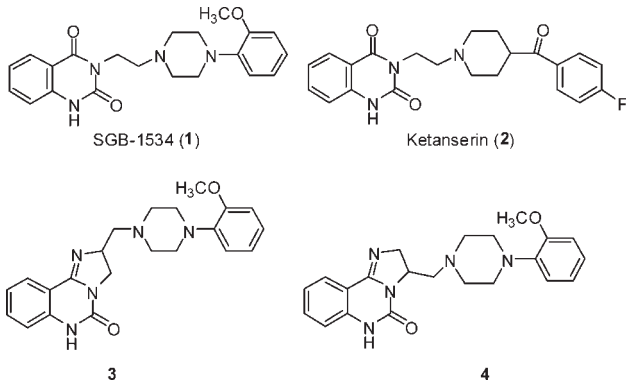


Figure 1. Some biologically active quinazolinones.

Table 1

Optimization for the reductive cyclization reaction.

Entry	Temperature (°C)	Solvent	Ratio[a]	Isolated yield (%)
1	rt	CH_3CN	1:7	0
2	40	CH_3CN	1:7	0
3	60	CH_3CN	1:7	60
4	Reflux	CH_3CN	1:7	81
5	Reflux	CH_3CN	1:3	37
6	Reflux	CH_3CN	1:4	67
7	Reflux	CH_3CN	1:5	70
8	Reflux	CH_3CN	1:6	75
9	Reflux	CH_3CN	1:8	82
10	Reflux	THF	1:7	42
11	Reflux	$CHCl_3$	1:7	0

[a] Ratio of **1** and $SnCl_2{\cdot}2H_2O$ system.

evaluate the influence of the ratio of **1a**:$SnCl_2{\cdot}2H_2O$, the reaction was carried out in acetonitrile using a 1:3–1:8 ratio of **1a**:$SnCl_2{\cdot}2H_2O$ (Table 1, entries 5, 6, 7, 8, 4, 9), leading to **3a** in 37%, 67%, 70%, 75%, 81%, and 82% yields, respectively. We concluded the best ratio of **1a**:$SnCl_2{\cdot}2H_2O$ was 1:7. Moreover, different organic solvents were further investigated as shown in Table 1; we concluded that acetonitrile was the best solvent for this reaction.

Having established an optimal condition for the protocol, we performed a more detailed examination of the substrates. Thus, the behavior of a variety of substrates, which include different 2-(2-nitrophenyl)-1*H*-imidazoles as well as different isothiocyanates or isocyanates was examined.

First of all, we performed the reaction of a variety of 2-(2-nitrophenyl)-1*H*-imidazoles **1** and isothiocyanates **2** via $SnCl_2{\cdot}2H_2O$ system (Scheme 2, Table 2).

As shown in Table 2, it can be seen that this protocol can be applied to isothiocyanates with either electron-withdrawing groups (such as halide groups) or electron-donating groups (such as alkyl groups) under the same conditions. Furthermore, it was particularly noteworthy that the effects of substituted 2-(2-nitrophenyl)-1*H*-imidazoles were also investigated. 4-fluorophenyl and 4-methoxyphenyl substitution can also give moderate to good yields.

A second part of the research was designed to synthesize imidazo[1,2-*c*]quinazolin-5(6*H*)-ones *via* the novel reductive cyclization of 2-(2-nitrophenyl)-1*H*-imidazoles with isocyanates under the same reaction conditions (Scheme 3, Table 3).

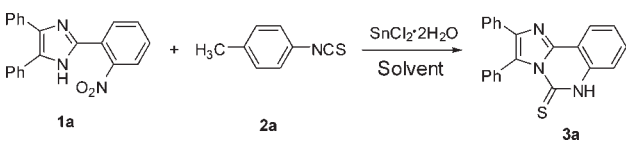
Similarly, aryl isocyanates containing electron-donating and electron-withdrawing substituents were reacted well with 2-(2-nitrophenyl)-1*H*-imidazoles, therefore, we can conclude that the electronic nature of the substituents has no significant effect on this reaction. Meanwhile, it was found that isocyanates showed better reactivity trends than isothiocyanates.

A plausible mechanistic pathway to products **3** and **5** is illustrated in Scheme 4, although the details are still unclear. In the initial step, 2-(2-nitrophenyl)-1*H*-imidazoles **1** are reduced by tin(II) chloride to **A**. The amine compounds **A** then reacted with isothiocyanates or isocyanates to give intermediate **B**. Intermediate **C** was formed by attack of the amino group onto the the central carbon atom of the urea or thiourea. Finally, products **3** and **5** were obtained by eliminating a amine molecule.

The structures of products **3** and **5** were confirmed by IR, ^{1}H NMR, and HRMS.

In summary, a series of imidazo[1,2-*c*]quinazoline-5(6*H*)-thiones and imidazo[1,2-*c*]quinazolin-5(6*H*)-ones were synthesized *via* $SnCl_2{\cdot}2H_2O$ induced reductive cyclization of isothiocyanates and isocyanates with 2-(2-nitrophenyl)-1*H*-imidazoles, respectively. The new method has advantages, such as easily accessible starting materials, convenient manipulation, and moderate to high yields.

Scheme 1



Scheme 2

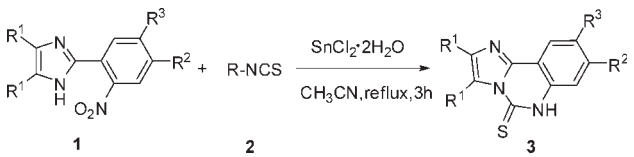


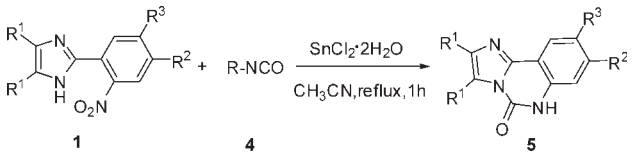
Table 2

Synthesis of imidazo[1,2-*c*]quinazoline-5(6*H*)-thiones.

Compound	R^1	R^2	R^3	R	Yield[a] (%)
3a	4-$CH_3OC_6H_4$	H	Cl	4-$CH_3C_6H_4$	78
3b	C_6H_5	H	Cl	C_6H_5	90
3c	C_6H_5	H	H	4-$CH_3C_6H_4$	81
3d	4-$CH_3C_6H_4$	H	H	4-$CH_3C_6H_4$	88
3e	4-FC_6H_4	H	H	4-ClC_6H_4	81
3f	4-$CH_3OC_6H_4$	H	H	3-$CH_3C_6H_4$	84

aIsolated yield.

Scheme 3



EXPERIMENTAL

Commercial solvents and reagents were used as received. Melting points were uncorrected. IR spectra were recorded on Varian F-1000 spectrometer in KBr with absorptions in cm^{-1}. ^1H NMR was determined on Varian-400 MHz spectrometer in DMSO-d_6 solution. *J* values are in Hz. Chemical shifts are expressed in ppm downfield from internal standard TMS. HRMS data were obtained using micromа GCT-TOF instrument.

General procedure for synthesis of imidazo[1,2-*c*]quinazoline-5(6*H*)-thione 3. A solution of 2-(2-nitrophenyl)-1H-imidazoles (0.5 mmol), isothiocyanates (0.5 mmol), and $SnCl_2{\cdot}2H_2O$ (3.5 mmol) in CH_3CN (5 mL) was stirred at reflux for 3 h. After this period, the TLC analysis of the mixture showed the reaction to be completed. The mixture was quenched with 3% HCl (10 mL) and filtered to yield a crude product, which was purified *via* silica gel column chromatography using acetone/petroleum ether (1:9, v/v) as eluent to furnish pure products in 78–90% yield.

9-Chloro-2,3-bis(4-methoxyphenyl)imidazo[1,2-c] quinazoline-5(6H)-thione (3a). This compound was obtained as solid with mp 284–286°C; IR(KBr):ν: 3172, 3109, 3006, 2960, 1616, 1533, 1520, 1490, 1472, 1389, 1369, 1312, 1291, 1245, 1171, 1134, 1083, 1034, 984, 875, 840, 816, 743, 696 cm^{-1}. ^1H NMR (DMSO-d_6): 3.73(s, 3H, OCH_3), 3.84 (s, 3H, OCH_3), 6.85 (d, J = 8.4 Hz, 2H, ArH),6.97 (d, J = 7.6 Hz, 2H, ArH), 7.30 (d, J = 8.0 Hz, 2H, ArH), 7.46 (d, J = 8.4 Hz, 2H, ArH), 7.56 (d, J = 8.4 Hz, 1H, ArH), 7.67 (d, J = 8.8 Hz, 1H, ArH), 8.26 (s, 1H, ArH), 13.38 (s, 1H, NH).

HRMS [Found: *m*/*z* 447.0806 (M^+), calcd for $C_{24}H_{18}N_3O_2S^{35}Cl$: M, 447.0808].

9-Chloro-2,3-diphenylimidazo[1,2-c]quinazoline-5(6H)-thione (3b). This compound was obtained as solid with mp 284–287°C; IR (KBr) ν: 3156, 3081, 3001, 2938, 1601, 1526, 1473, 1443, 1367, 1303, 1265, 1172, 1136, 1088, 875, 819, 777, 759, 694 cm^{-1}. ^1H NMR (DMSO-d_6): 7.25–7.27 (m, 3H, ArH), 7.40–7.44 (m, 4H, ArH), 7.47–7.50 (m, 3H, ArH), 7.57 (d, J = 8.8 Hz, 1H, ArH), 7.69 (dd, J_1 = 2.4 Hz, J_2 = 8.4 Hz, 1H, ArH), 8.28 (d, J = 2.0, 1H, ArH), 13.45 (s, 1H, NH).

HRMS [Found: *m*/*z* 387.0594 (M^+), calcd for $C_{22}H_{14}N_3S^{35}Cl$: M, 387.0597].

2,3-Diphenylimidazo[1,2-c]quinazoline-5(6H)-thione (3c). This compound was obtained as solid with mp 278–280°C (ref. 7; 283–288°C); IR (KBr) ν: 3160, 3093, 3012, 2957, 1626, 1530, 1481, 1443, 1398, 1375, 1324, 1287, 1174, 1132, 1065, 753, 695 cm^{-1}. ^1H NMR (DMSO-d_6): 7.25–7.26 (m, 3H, ArH), 7.40–7.43 (m, 5H, ArH), 7.48–7.49 (m, 3H, ArH), 7.58 (d, J = 8.0 Hz, 1H, ArH), 7.65 (t, J = 7.2 Hz, 1H, ArH), 8.35 (d, J = 7.6 Hz, 1H, ArH), 13.36 (s, 1H, NH).

HRMS [Found: *m*/*z* 353.0985 (M^+), calcd for $C_{22}H_{15}N_3S$: M, 353.0987].

2,3-Dip-tolylimidazo[1,2-c]quinazoline-5(6H)-thione (3d). This compound was obtained as solid with mp 281–282°C; IR (KBr) ν: 3169, 3102, 3018, 2959, 1653, 1635, 1559, 1530, 1490, 1477, 1395, 1374, 1322, 1283, 1172, 1131, 825, 751, 696 cm^{-1}. ^1H NMR (DMSO-d_6): 2.25 (s, 3H, CH_3), 2.40 (s, 3H, CH_3), 7.07 (d, J = 8.0 Hz, 2H, ArH), 7.19 (d, J = 7.6 Hz, 2H, ArH), 7.27 (d, J = 8.0 Hz, 2H, ArH), 7.38 (d, J = 8.0 Hz, 2H, ArH), 7.46 (t, J = 7.2 Hz, 1H, ArH), 7.56 (d, J =

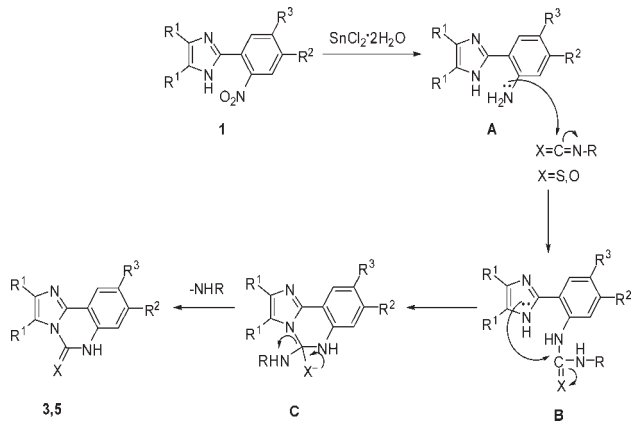
Table 3

Synthesis of imidazo[1,2-*c*]quinazolin-5(6*H*)-ones.

Compound	R^1	R^2	R^3	R	Yield[a] (%)
5a	C_6H_5	H	H	4-$CH_3C_6H_4$	89
5b	C_6H_5	H	H	4-ClC_6H_4	82
5c	4-$CH_3C_6H_4$	H	H	3-ClC_6H_4	73
5d	4-$CH_3OC_6H_4$	H	Cl	C_6H_{11}	85
5e	C_6H_5	3,4-OCH_2O		3-$CH_3C_6H_4$	85

aIsolated yield.

Scheme 4



8.0 Hz, 1H, ArH), 7.63 (t, J = 7.2 Hz, 1H, ArH), 8.33 (d, J = 8.0 Hz, 1H, ArH), 13.31 (s, 1H, NH).

HRMS [Found: m/z 381.1301 (M^+), calcd for $C_{24}H_{19}N_3S$: M, 381.1300].

2,3-Bis(4-fluorophenyl)imidazo[1,2-c]quinazoline-5(6H)-thione (3e). This compound was obtained as solid with mp 288–290°C; IR (KBr): ν: 3169, 3105, 3014, 2960, 1629, 1533, 1515, 1492, 1396, 1374, 1322, 1271, 1222, 1163, 1130, 844, 818, 782, 747, 698 cm^{-1}. ^1H NMR (DMSO-d_6): 7.15 (t, J = 8.8 Hz, 2H, ArH), 7.24 (t, J = 8.8 Hz, 2H, ArH), 7.45–7.53 (m, 5H, ArH), 7.58 (d, J = 8.4, 1H, ArH), 7.66 (t, J = 7.6 Hz, 1H, ArH), 8.34 (d, J = 8.0 Hz, 1H, ArH), 13.39 (s, 1H, NH).

HRMS [Found: m/z 389.0794 (M^+), calcd for $C_{22}H_{13}N_3F_2S$: M, 389.0798].

2,3-Bis(4-methoxyphenyl)imidazo[1,2-c]quinazoline-5(6H)-thione (3f). This compound was obtained as solid with mp 258–260°C; IR (KBr) ν: 3170, 3104, 3008, 2957, 2834, 1614, 1536, 1496, 1476, 1377, 1326, 1283, 1249, 1176, 1035, 834, 754, 697 cm^{-1}. ^1H NMR (DMSO-d_6): 3.73 (s, 3H, OCH_3), 3.84 (s, 3H, OCH_3), 6.85 (d, J = 8.8 Hz, 2H, ArH), 6.96 (d, J = 8.4 Hz, 2H, ArH), 7.30 (d, J = 8.0 Hz, 2H, ArH), 7.44–7.47 (m, 3H, ArH), 7.56 (d, J = 8.4 Hz, 1H, ArH), 7.63 (t, J = 7.6 Hz, 1H, ArH), 8.33 (d, J = 7.6 Hz, 1H, ArH), 13.28 (s, 1H, NH).

HRMS [Found: m/z 413.1196 (M^+), calcd for $C_{24}H_{19}N_3O_2S$: M, 413.1198].

General procedure for synthesis of imidazo[1,2-*c*]quinazolin-5(6*H*)-one 5. A solution of 2-(2-nitrophenyl)-1*H*-imidazoles (0.5 mmol), isocyanates (0.5 mmol), and $SnCl_2{\cdot}2H_2O$ (3.5 mmol) in CH_3CN (5 mL) was stirred at reflux for 1 h. After this period, the TLC analysis of the mixture showed the reaction to be completed. The mixture was quenched with 3% HCl (10 mL) and filtered and the crude product was purified by recrystallization from 95% ethanol and DMF.

2,3-Diphenylimidazo[1,2-c]quinazolin-5(6H)-one (5a). This compound was obtained as solid with mp > 300°C (ref. 9; >300°C); IR (KBr): ν: 3215, 3164, 3103, 3053, 2926, 1706, 1597, 1555, 1481, 1443, 1379, 1336, 1216, 1109, 803, 780, 749, 696 cm^{-1}. ^1H NMR (DMSO-d_6): 7.21–7.26 (m, 4H, ArH), 7.30–7.35 (m, 2H, ArH), 7.42–7.44 (m, 6H, ArH), 7.54 (t, J = 7.2 Hz, 1H, ArH), 8.23 (d, J = 7.6 Hz, 1H, ArH), 11.72 (s, 1H, NH).

2,3-Diphenylimidazo[1,2-c]quinazolin-5(6H)-one (5b). This compound was obtained as solid with mp > 300°C (ref. 9; >300°C); IR (KBr): ν: 3215, 3164, 3103, 3053, 2926, 1706, 1597, 1555, 1481, 1443, 1379, 1336, 1263, 1216, 1110, 803, 780, 749, 696 cm^{-1}. ^1H NMR (DMSO-d_6): 7.19–7.25 (m, 3H, ArH), 7.29–7.35 (m, 2H, ArH), 7.41–7.44 (m, 7H, ArH), 7.53 (t, J = 7.2 Hz, 1H, ArH), 8.23 (t, J = 6.8 Hz, 1H, ArH), 11.71 (s, 1H, NH).

2,3-Dip-tolylimidazo[1,2-c]quinazolin-5(6H)-one (5c). This compound was obtained as solid with mp > 300°C (ref. 9; > 300°C); IR (KBr): ν: 3224, 3167, 2933, 2875, 1706, 1596, 1551, 1491, 1399, 1344, 1207, 961, 819, 745, 693 cm^{-1}. ^1H NMR (DMSO-d_6): 2.26 (s, 3H, CH_3), 2.40 (s, 3H, CH_3), 7.08 (d, J = 8.0 Hz, 2H, ArH), 7.23 (d, J = 8.4 Hz, 2H, ArH), 7.31 (d, J = 8.0 Hz, 2H, ArH), 7.33–7.37 (m, 4H, ArH), 7.55 (t, J = 7.6 Hz, 1H, ArH), 8.24 (d, J = 7.6 Hz, 1H, ArH), 11.71 (s, 1H, NH).

9-Chloro-2,3-bis(4-methoxyphenyl)imidazo[1,2-c] quinazolin-5(6H)-one (5d). This compound was obtained as solid with mp > 300°C (ref. 9; > 300°C); IR (KBr): ν: 3210, 3092, 2906, 2835, 1701, 1614, 1555, 1521, 1491, 1367, 1331, 1288, 1247, 1171, 1038, 829 cm^{-1}. ^1H NMR (DMSO-d_6): 3.73 (s, 3H, OCH_3), 3.83 (s, 3H, OCH_3), 6.85 (d, J = 8.8 Hz, 2H, ArH), 6.99 (d, J = 8.8 Hz, 2H, ArH), 7.35 (d, J = 8.8 Hz, 3H, ArH), 7.42 (d, J = 8.8 Hz, 2H, ArH), 7.58 (dd, J_1 = 2.4 Hz, J_2 = 8.8 Hz, 1H, ArH), 8.16 (d, J = 2.4 Hz, 1H, ArH), 11.81 (s, 1H, NH).

2,3-Diphenyl-[1,3]dioxolo[4,5-g]imidazo[1,2-c] quinazolin-5(6H)-one (5e). This compound was obtained as solid with mp > 300°C; IR (KBr): ν: 3251, 3196, 3088, 3017, 2858, 1719, 1659, 1565, 1473, 1354, 1310, 1270, 1200, 1039, 936, 862, 801, 748, 706, 667 cm^{-1}. ^1H NMR (DMSO-d_6): 6.16 (s, 2H, OCH_2O), 6.87 (s, 1H, ArH), 7.22–7.27 (m, 3H, ArH), 7.43–7.45 (m, 7H, ArH), 7.62 (s, 1H, ArH), 11.62 (s, 1H, NH).

HRMS [Found: m/z 381.1124 (M^+), calcd for $C_{23}H_{15}N_3O_3$: M, 381.1113].

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