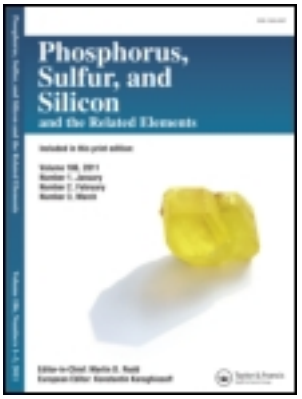

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An Efficient and Convenient Protocol for the Synthesis of Thiazolidin-4-Ones

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AN EFFICIENT AND CONVENIENT PROTOCOL FOR THE SYNTHESIS OF THIAZOLIDIN-4-ONES

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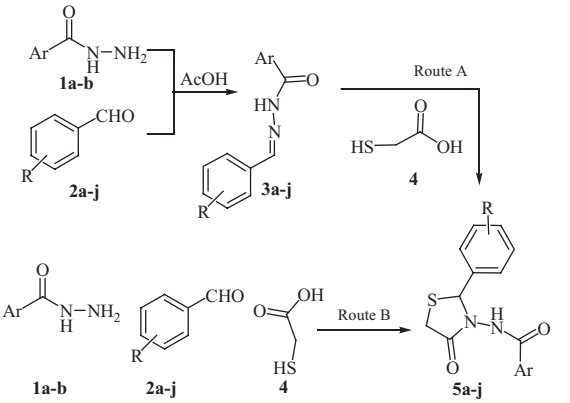
GRAPHICAL ABSTRACT



Abstract *Without a catalyst and under solvent-free conditions, 2,3-disubstituted-1,3-thiazolidin-4-one* ***5a–j*** *derivatives were synthesized efficiently via the three-component reaction of aryl hydrazide, aromatic aldehyde, and mercaptoacetic acid. All the newly synthesized compounds were confirmed by IR, ^{1}H NMR, and ^{13}C NMR spectroscopy and elemental analysis.*

Keywords Thiazolidin-4-one; multicomponent; isonicotinohydrazide; nicotinohydrazide; mercaptoacetic acid

INTRODUCTION

Synthetic modifications in which highly functionalized organic molecules can be produced from easily available substrates via atom-efficient reactions have special importance for the organic chemists, for example, in expeditious domino and multicomponent

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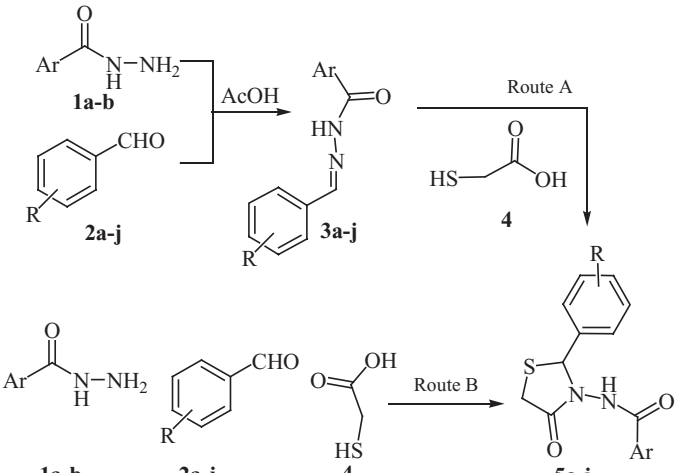
reactions (MCRs).[1] In these reactions, three, four, or five easily accessible components react to form a single product, which incorporates essentially all the carbon atoms of the starting materials.[2]

The Thiazolidin-4-one ring system is a core structure in various synthetic pharmaceutical compounds, displaying a broad spectrum of biological activities.[3–8] Several synthetic methods have been developed for the synthesis of thiazolidin-4-ones. The main synthetic routes to thiazolidin-4-ones involve cyclocondensation of azomethines (Schiff's base) with mercaptoacetic acid.[9] There are also reports of using chemical agents such as *N,N*-dicyclohexyl carbodiimide (DCC)[10] as desiccant to assist the formation of thiazolidinone derivatives. The use of ionic liquid,[11] Hunig's base,[12] and KSF Montmorillonite,[13] has also been reported to expedite the cyclocondensation of the azomethines and thioglycolic acid. However these methodologies suffer from one or more disadvantages such as costly dehydrating agents and require prolonged heating and tedious work-up. Therefore it was thought worthwhile to develop a new method for the cyclocondensation. In continuation of our effort to develop the synthesis of new heterocyclic using the reaction of azomethines with nucleophile,[14] we report, herein, multicomponent procedure for the synthesis of thiazolidinone compounds without the use of a catalyst.

RESULTS AND DISCUSSION

In the present study, we report our results on a new type of the MCR where three organic components react to form novel compounds. Two strategies were attempted for the synthesis of thiazolidin-4-ones **5a–j**. First, the synthesis of thiazolidin-4-ones was investigated through a two-step pathway (see Scheme 1, Route A). Initially, the *N*-benzylideneisonicotinohydrazide intermediates **3a–j** were obtained by refluxing the aryl hydrazide **1a,b** and aromatic aldehyde **2a–j** in acetic acid with excellent yields (85%–90%).



Ar= 3-pyridyl, 4-pyridyl

R= H, 4-Me, 3-Br, 2-Cl, 4-Cl, 2-NO_2, 3-NO_2, 4-NO_2

Scheme 1

Table 1 Thiazolidin-4-one **5a–j**

Products	R	Ar	Route A yield (%)	Route B yield (%)
5a	H	4-pyridyl	80	70
5b	4-CH_3	4-pyridyl	70	65
5c	3-Br	4-pyridyl	64	60
5d	2-Cl	4-pyridyl	90	90
5e	4-Cl	4-pyridyl	85	70
5f	2-NO_2	4-pyridyl	65	65
5g	3-NO_2	4-pyridyl	90	85
5h	4-NO_2	4-pyridyl	85	85
5i	3- NO_2	3-pyridyl	80	69
5j	4-NO_2	3-pyridyl	85	72

Subsequently, the thiazolidin-4-ones **5a–j** were synthesized from cyclocondensation reactions of the acyl hydrazones intermediates **3a–j** by using excess of mercaptoacetic acid **4**, without any solvent at 60°C (see Scheme 1, Route A). The results are summarized in Table 1.

We also carried out the synthesis of 2,3-disubstituted-1,3-thiazolidin-4-one **5a–j** that involves the one-pot, three-component condensation reactions of aryl hydrazide, different aromatic aldehydes, and large excess of thioglycolic acid under solvent-free condition (see Scheme 1, Route B). The initial reaction using isonicotinohydrazide **1a** and aldehyde **2a** at a ratio of 1:1.2, and excess of thioglycolic acid **4**, was stirred under 60°C. When the reaction was terminated [monitored by thin layer chromatography (TLC)], the expected product **5a** was afforded in a yield of 45%. The yield of **5a** increased remarkably with increasing temperature until 80°C.

Encouraged by the result obtained above, we extended this process to various aldehydes to gain more insight into this reaction (see Scheme 1, Route B). The typical results are summarized in Table 1. Aromatic aldehydes with substitutes carrying either electron-donating or electron-withdrawing groups reacted successfully and gave the products in good yields.

The ^{1}H NMR spectra of all the synthesized compounds reveal a singlet at $\delta =$ 5.90–6.30 ppm attributed to the resonance of the methine proton (N—CH—S). The appearance of the IR absorptions and ^{13}C-NMR signals, due to the carbonyl group of the synthesized compounds **5a–j**, clearly confirmed the formation of thiazolidin-4-ones.

CONCLUSION

In summary, we reported a novel and efficient method for the synthesis of thiazolidin-4-ones. The notable advantages of our work are as follows: (1) the reaction is performed under mild condition and more important is under solvent-free condition, (2) no catalyst is required for this reaction, (3) good yields of cyclization products without any byproduct, and (4) simple purification method.

EXPERIMENTAL

Purity of the compounds was checked by TLC using ethyl acetate/*n*-hexane (1:1 v/v) as an eluent. All products were characterized using IR, ^{1}H NMR, and ^{13}C NMR spectra.

All yields refer to isolated products. IR spectra were prepared on a Galaxy series FTIR 5000 spectrophotometer (Perkin Elmer) using KBr discs. NMR spectra were recorded on Bruker spectrophotometer (300 MHz) in dimethyl sulfoxide-d_6 (DMSO-d_6) using TMS as an internal standard. C, H, N, and S analyses were performed on a Vario EL III elemental analyzer.

General Procedure for the Synthesis of Thiazolidine-4-one 5a–j

Route A. A mixture of acylhydrazone **3a–j** (0.1 mol), mercaptoacetic acid (0.5 mL), was heated at 60°C under solvent-free conditions for 3 h. The progress of the reaction was monitored by TLC using *n*-hexane–ethyl acetate (1:1). After the completion of the reaction, the mixture was extracted with ethyl acetate and the organic layer was washed with 5% sodium bicarbonate solution and water. The organic layer was separated and dried over anhydrous sodium sulfate. From the organic extract, the solvent was removed under reduced pressure. The residue obtained was triturated with *n*-hexane (2 mL) and filtered to isolate the solid product, which was then washed with *n*-hexane (2 mL), and dried to give thiazolidin-4-one **5a**–**j**.

Route B. A mixture of acylhydrazides **1a,b** (1 mmol), and appropriate aromatic aldehyde (1.2 mmol), mercaptoacetic acid (0.5 mL), was stirred at 80 °C in an oil bath for 7 h. Then, the reaction mixture was extracted with ethyl acetate and the organic layer was washed with 5% $NaHCO_3$ solution/water and dried over anhydrous Na_2SO_4. The crude solid obtained on evaporation of the solvent under reduced pressure was recrystallized from ethylacetat/*n*-xexane to furnish a crystalline solid **5a–j**.

N-(4-Oxo-2-phenylthiazolidin-3-yl)isonicotinamide (5a, Table 1)

IR (KBr): $\upsilon = 3108$ (NH), 3035 (aromatic CH stretch.), 2995 (aliphatic CH stretch.), 1716 (C=O), 1682 (C=O), 1616 (C=N), 1524 (C=C) cm^{-1}, ^{1}H NMR (DMSO-d_6) δ (ppm): 3.80 (d, 1H, CH_2, $J = 16.1$ Hz), 3.91(d, 1H, CH_2, $J = 16.2$ Hz), 5.91 (s, 1H, S−CH−N), 7.36–7.47 (m, 5H, $H_{arom.}$), 7.60 (d, 2H, $H_{arom.}$, $J = 5.3$ Hz), 8.69 (d, 2H, $H_{arom.}$, $J = 5.5$ Hz), 11.10 (s, 1H, D_2O exchange, NH), ^{13}C NMR (DMSO-d_6) δ (ppm): 29.8 (CH_2), 62.0 (CH), 121.7, 128.2, 129.1, 129.6, 138.4, 138.9, 150.9, 164.3 (C=O), 169.6 (C=O), Anal. Calcd. for: $C_{15}H_{13}N_3O_2S$: C, 60.18; H, 4.38; N, 14.04; S, 10.71. Found: C, 60.02; H, 4.31; N, 13.96; S, 10.64.

N-(4-Oxo-2-p-tolylthiazolidin-3-yl)isonicotinamide (5b, Table 1)

IR (KBr): $\upsilon = 3103$ (NH), 3020 (aromatic CH stretch.), 2881 (aliphatic CH stretch.), 1721 (C=O), 1680 (C=O), 1602 (C=N), 1514 (C=C) cm^{-1}, ^{1}H NMR (DMSO-d_6) δ (ppm): 2.29 (s, 3H, CH_3), 3.83 (d, 1H, CH2, $J = 15.9$ Hz), 3.95 (d, 1H, CH_2, $J = 16.1$ Hz), 5.90 (s, 1H, S-CH-N), 7.19 (d, 2H, $H_{arom.}$, $J = 7.6$ Hz), 7.38 (d, 2H, $H_{arom.}$, $J = 7.6$ Hz), 7.63 (d, 2H, $H_{arom.}$, $J = 4.5$ Hz), 8.72 (d, 2H, $H_{arom.}$, $J = 4.6$ Hz), 11.03 (s, 1H, D_2O exchange, NH), ^{13}C NMR (DMSO-d_6) δ (ppm): 21.3 (CH_3), 29.9 (CH_2), 62.0 (CH), 121.77, 128.3, 129.6, 135.3, 138.9, 139.1, 150.9, 164.3 (C=O), 169.6 (C=O), Anal. Calcd. for: $C_{16}H_{15}N_3O_2S$: C, 61.32; H, 4.82; N, 13.41; S, 10.23. Found: C, 61.22; H, 4.76; N, 13.29; S, 10.09.

N-(2-(3-Bromophenyl)-4-oxothiazolidin-3-yl)isonicotinamide (5c, Table 1)

IR (KBr): $\upsilon = 3110$ (NH), 3020 (aromatic CH stretch.), 2924 (aliphatic CH stretch.), 1718 (C=O), 1685 (C=O), 1523 (C=C) cm^{-1}, ^{1}H NMR (DMSO-d_6) δ (ppm): 3.83 (d, 1H, CH_2, $J = 15.8$ Hz), 3.98 (d, 1H, CH_2, $J = 15.6$ Hz), 5.94 (s, 1H, S-CH-N), 7.35–7.64

(m, 5H, $H_{arom.}$), 7.73 (s, 1H, $H_{arom.}$), 8.74 (d, 2H, $H_{arom.}$, $J = 5.5$ Hz), 11.10 (s, 1H, D_2O exchange, NH), ^{13}C NMR (DMSO-d_6) δ (ppm): 29.6 (CH_2), 61.2 (CH), 121.7, 122.3, 127.4, 130.7, 131.2, 132.4, 138.9, 141.6, 151.0, 164.4 (C=O), 169.5 (C=O), Anal. Calcd. for: $C_{15}H_{12}ClN_3O_2S$: C, 47.63; H, 3.20; N, 11.11; S, 8.48. Found: C, 47.50; H, 3.16; N, 10.98; S, 8.30.

N-(2-(2-Chlorophenyl)-4-oxothiazolidin-3-yl)isonicotinamide (5d, Table 1)

IR (KBr): υ = IR (KBr): υ (cm^{-1}): 3107 (NH), 3040 (aromatic CH stretch.), 2879 (aliphatic CH stretch.), 1722 (C=O), 1682 (C=O), 1602 (C=N), 1529 (C=C) cm^{-1}, ^1H NMR (DMSO-d_6) δ (ppm): 3.84 (d, 1H, CH_2, $J = 15.9$ Hz), 3.99 (d, 1H, CH_2, $J = 15.8$ Hz), 6.28 (s, 1H, S-CH-N), 7.40–7.47 (m, 3H, $H_{arom.}$), 7.59–7.72 (m, 3H, $H_{arom.}$), 8.73 (d, 2H, $H_{arom.}$, $J = 5.6$ Hz), 11.16 (s, 1H, D_2O exchange, NH), ^{13}C NMR (DMSO-d_6) δ (ppm): 29.3 (CH_2), 58.7 (CH), 121.8, 128.4, 129.0, 130.1, 131.0, 132.7, 136.2, 138.9, 151.0, 164.6 (C=O), 169.8 (C=O). Anal. Calcd. for: $C_{15}H_{12}ClN_3O_2S$: C, 53.97; H, 3.62; N, 12.59; S, 9.61. Found: C, 53.69; H, 3.52; N, 12.41; S, 9.50.

N-(2-(4-Chlorophenyl)-4-oxothiazolidin-3-yl)isonicotinamide (5e, Table 1)

IR (KBr): $\upsilon = 3120$ (NH), 3010 (aromatic CH stretch.), 2992 (aliphatic CH stretch.), 1720 (C=O), 1680 (C=O), 1610 (C=N) cm^{-1}, ^1H NMR (DMSO-d_6) δ (ppm): 3.85 (d, 1H, CH_2, $J = 16.2$ Hz), 3.98 (d, 1H, CH_2, $J = 16.0$ Hz), 5.94 (s, 1H, S-CH-N), 7.45–7.52 (m, 4H, $H_{arom.}$), 7.63 (d, 2H, $H_{arom.}$, $J = 5.6$ Hz), 8.72 (d, 2H, $H_{arom.}$, $J = 5.5$ Hz), 10.96 (s, 1H, D_2O exchange, NH), ^{13}C NMR (DMSO-d_6) δ (ppm): 29.7 (CH_2), 61.3 (CH), 121.8, 129.1, 130.2, 134.1, 137.7, 138.9, 151.0, 164.3 (C=O), $C_{15}H_{12}ClN_3O_2S$: C, 53.97; H, 3.62; N, 12.59; S, 9.61. Found: C, 53.72; H, 3.54; N, 12.40; S, 9.54.

N-(2-(4-Nitrophenyl)-4-oxothiazolidin-3-yl)isonicotinamide (5f, Table 1)

IR (KBr): $\upsilon = 3120$ (NH), 3054 (aromatic CH stretch.), 2941 (aliphatic CH stretch.), 1720 (C=O), 1688 (C=O), 1520 (C=C) cm^{-1}, ^1H NMR (DMSO-d_6) δ (ppm): 3.78 (d, 1H, CH_2, $J = 15.5$ Hz), 3.96 (d, 1H, CH_2, $J = 15.4$ Hz), 6.30 (s, 1H, S-CH-N), 7.42–7.66 (m, 3H, $H_{arom.}$), 7.88 (br, 2H, $H_{arom.}$), 8.07 (s, 1H, $H_{arom.}$), 8.72 (d, 2H, $H_{arom.}$, $J = 5.4$ Hz), 11.20 (s, 1H, D_2O exchange, NH), ^{13}C NMR (DMSO-d_6) δ (ppm): 29.6 (CH_2), 57.7 (CH), 121.9, 125.6, 127.9, 128.2, 130.2, 131.6, 135.3, 147.6, 150.8, 164.5 (C=O), 170.0 (C=O), Anal. Calcd. for: $C_{15}H_{12}N_4O_4S$: C, 52.32; H, 3.51; N, 16.27; S, 9.31. Found: C, 52.21; H, 3.45; N, 16.07; S, 9.14.

N-(2-(3-Nitrophenyl)-4-oxothiazolidin-3-yl)isonicotinamide (5g, Table 1)

IR (KBr): $\upsilon = 3115$ (NH), 3034 (aromatic CH stretch.), 2931 (aliphatic CH stretch.), 1715 (C=O), 1685 (C=O), 1521 (C=C) cm^{-1}, ^1H NMR (DMSO-d_6) δ (ppm): 3.83 (d, 1H, CH_2, $J = 15.5$ Hz), 3.99 (d, 1H, CH_2, $J = 15.7$ Hz), 6.14 (s, 1H, S-CH-N), 7.68 (d, 2H, $H_{arom.}$, $J = 7.5$ Hz), 7.92–8.00 (m, 1H, $H_{arom.}$), 8.22 (br, 1H, $H_{arom.}$), 8.32 (s, 1H, $H_{arom.}$), 8.41 (s, 1H, $H_{arom.}$), 8.70 (d, 2H, $H_{arom.}$, $J = 5.6$ Hz), 11.18 (br, 1H, D_2O exchange, NH), ^{13}C NMR (DMSO-d_6) δ (ppm): 30.4 (CH_2), 61.1 (CH), 121.7, 123.1, 124.4 130.7, 135.0, 138.8, 141.3, 148.3, 150.9, 164.4 (C=O), 169.5 (C=O), Anal. Calcd. for: $C_{15}H_{12}N_4O_4S$: C, 52.32; H, 3.51; N, 16.27; S, 9.31. Found: C, 52.18; H, 3.47; N, 16.08; S, 9.21.

N-(2-(4-Nitrophenyl)-4-oxothiazolidin-3-yl)isonicotinamide (5h, Table 1)

IR (KBr): $\upsilon = 3115$ (NH), 3078 (aromatic CH stretch.), 2982 (aliphatic CH stretch.), 1722 (C=O), 1676 (C=O), 1601 (C=N), 1521, 1346 (NO_2) cm^{-1}, ^1H NMR (DMSO-d_6) δ (ppm): 3.87 (d, 1H, CH2, $J = 16.0$ Hz), 4.05 (d, 1H, CH_2, $J = 16.1$ Hz), 6.09 (s, 1H, S-CH-N), 7.64 (d, 2H, $H_{arom.}$, $J = 5.6$ Hz), 7.81 (d, 2H, $H_{arom.}$, $J = 8.6$ Hz), 8.25 (d, 2H, $H_{arom.}$, $J = 8.5$ Hz), 8.73 (d, 2H, $H_{arom.}$, $J = 5.6$ Hz), 11.16 (s, 1H, D_2O exchange, NH), ^{13}C NMR (DMSO-d_6) δ (ppm): 29.6 (CH_2), 60.9 (CH), 121.8, 124.3, 129.5, 138.7, 146.6, 148.2, 151.0, 164.4 (C=O), 169.5 (C=O), Anal. Calcd. for: $C_{15}H_{12}N_4O_4S$: C, 52.32; H, 3.51; N, 16.27; S, 9.31. Found: C, 52.08; H, 3.36; N, 16.03; S, 9.07.

N-(2-(3-Nitrophenyl)-4-oxothiazolidin-3-yl)nicotinamide (5i, Table 1)

IR (KBr): $\upsilon = 3115$ (NH), 3064 (aromatic CH stretch.), 2944 (aliphatic CH stretch.), 1718 (C=O), 1680 (C=O), 1551 (C=C) cm^{-1}, ^1H NMR (DMSO-d_6) δ (ppm): 3.85 (d, 1H, CH_2, $J = 16.1$ Hz), 4.10 (d, 1H, CH_2, $J = 16.00$ Hz), 6.13 (s, 1H, S-CH-N), 7.48 (d, d 1H, $H_{arom.}$, $J = 7.4$, 4.9 Hz), 7.66–7.72 (m 1H, $H_{arom.}$, $J = 7.8$, 4.5 Hz), 8.00 (d, 1H, $H_{arom.}$, $J = 7.5$ Hz), 8.10 (d, 1H, $H_{arom.}$, $J = 7.8$ Hz), 8.22 (d, 1H, $H_{arom.}$, $J = 7.6$ Hz), 8.40 (br, 1H, $H_{arom.}$), 8.71 (d, 1H, $H_{arom.}$, $J = 4.0$ Hz), 8.88 (s, 1H, H_{arom}), 11.12 (s, 1H, D_2O exchange, NH), ^{13}C NMR (DMSO-d_6) δ (ppm): 29.7 (CH_2), 61.2 (CH), 122.9, 124.1, 124.4, 127.6, 130.7, 135.0, 135.8, 141.5, 148.3, 148.9, 153.4, 164.5 (C=O), 169.6 (C=O). Anal. Calcd. for: $C_{15}H_{12}N_4O_4S$: C, 52.32; H, 3.51; N, 16.27; S, 9.31. Found: C, 52.24; H, 3.48; N, 16.11; S, 9.20.

N-(2-(4-Nitrophenyl)-4-oxothiazolidin-3-yl)nicotinamide (5j, Table 1)

IR (KBr): $\upsilon = 3110$ (NH), 3034 (aromatic CH stretch.), 2928 (aliphatic CH stretch.), 1718 (C=O), 1686 (C=O), 1532 (C=C) cm^{-1}, ^1H NMR (DMSO-d_6) δ (ppm): 3.83 (d, 1H, CH_2, $J = 15.9$ Hz), 3.99 (d, 1H, CH_2, $J = 16.00$ Hz), 6.10 (s, 1H, S-CH-N), 7.50 (d, d 1H, $H_{arom.}$, $J = 7.7$, 4.9 Hz), 7.82 (d, 2H, $H_{arom.}$, $J = 8.4$ Hz), 8.10 (d, 1H, $H_{arom.}$, $J = 7.7$ Hz), 8.24 (d, 2H, $H_{arom.}$, $J = 8.3$ Hz), 8.71 (d, 2H, $H_{arom.}$, $J = 4.4$ Hz), 8.90 (s, 1H, H_{arom}), 11.10 (br, 1H, D_2O exchange, NH), ^{13}C NMR (DMSO-d_6) δ (ppm): 29.7 (CH_2), 61.1 (CH), 124.2, 127.5, 129.2, 129.5, 135.9, 146.6, 148.2, 148.9, 153.4, 164.5 (C=O), 169.6 (C=O), Anal. Calcd. for: $C_{15}H_{12}N_4O_4S$: C, 52.32; H, 3.51; N, 16.27; S, 9.31. Found: C, 52.04; H, 3.45; N, 16.16; S, 9.14.

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