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An Efficient and Practical Method for the Synthesis of 1-(2,6-Difluorobenzoyl)-3-(2-alkyl-3-oxopyridazin-4-yl)ureas as Potential Chitin Synthesis Inhibitors

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Summary. A mild and efficient method for the synthesis of 4-amino-3(2*H*)-pyridazinones from their corresponding 4,5-dichloropyridazinones under microwave-assisted conditions is described. A series of novel chitin synthesis inhibitors, benzoylphenylureas containing the 3(2*H*)-pyridazinone, were synthesized. The biological activity of these target compounds was evaluated.

Keywords. Bioorganic chemistry; Heterocycles; Microwave; Insecticidal activity.

Introduction

In the past decades benzoylphenylureas have attracted considerable interest because of their insecticidal and antitumor activities [1]. In pesticides chemistry, these compounds are generally recognized as insect growth regulators that interfere with chitin synthesis causing death or abortive development [2]. Results from structure-activity relationship studies of benzoylphenylureas revealed that the basic 2,6-dihalo configuration in the benzoylurea moiety is critical to the activity [3, 4] (Fig. 1). Now, more than ten members of this class of commercial pesticides containing the *N*-(2,6-difluorobenzoyl)urea moiety have been manufactured and are used widely in crop protection [5].

On the other hand, the 3(2*H*)-pyridazinone core has been utilized as a key template in the search of new medicines and agrochemicals [6, 7]. *Canada et al.*

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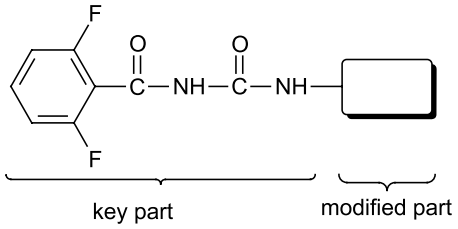


Fig. 1. *N*-2,6-Difluorobenzoylurea moiety

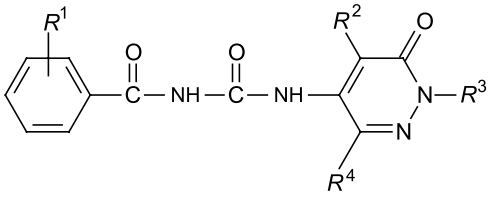


Fig. 2. Some bioactive benzoyl oxopyridazin-4-yl ureas

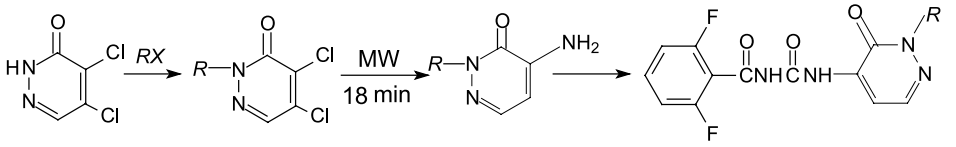
have attached the 5-position of 3(2*H*)-pyridazinone core to the benzoylurea moiety (Fig. 2), and it has been found that these compounds exhibited a significant insecticidal activity on the southern armyworm [8]. Although the key intermediate, 5-amino-3(2*H*)-pyridazinone, has been prepared by a traditional method [8], the synthesis of 4-amino-3(2*H*)-pyridazinone seems to be difficult [9], therefore, there are no reports on the introduction of the 4-position of 3(2*H*)-pyridazinone to benzoylurea to date.

In our research group, we have been interested in studying the design, synthesis, and biological activity of compounds containing the 3(2*H*)-pyridazinone nucleus [10, 11]. In our earlier research, we briefly reported for the first time a novel approach to 4-amino-3(2*H*)-pyridazinone *via* an unusual direct amination of 4,5-dichloropyridazinones with hydrazine hydrate under mild conditions [12].

To extend our previous work, we developed as a first step a new microwave-enhanced synthesis to obtain the key intermediates 4-amino-3(2*H*)-pyridazinones in a short reaction time, and then in the second step we combined the bioactive units of the *N*-(2,6-difluorobenzoyl)urea moiety and the 4-position of the 3(2*H*)-pyridazinone core to synthesize novel 1-(2,6-difluorobenzoyl)-3-(2-alkyl-3-oxopyridazin-4-yl) ureas in order to find new compounds with higher insecticidal activity.

Results and Discussion

The title compounds were prepared as shown in Scheme 1. The starting material 4,5-dichloro-3(2*H*)-pyridazinones were prepared by treating mucochloric acid with semicarbazide hydrochloric acid in 50% ethanol [13]. 2-*tert*-Butyl-4,5-dichloro-3(2*H*)-pyridazinone (**1f**) and 2-phenyl-4,5-dichloro-3(2*H*)-pyridazinone (**1h**) were prepared by treating mucochloric acid with *tert*-butylhydrazine hydrochloride and phenylhydrazine hydrochloride [13]. Because the alkylhydrazine hydrochlorides were commercially unavailable, the other 2-alkyl-4,5-dichloro-3(2*H*)-pyridazinones **1a**–**1e** and **1g** were prepared by conventional alkylation [14]. The key intermediates



1a–3a : $R = CH_3$ **1b–3b** : $R = CH_2CH_3$ **1c–3c** : $R = CH_2CH_2CH_3$ **1d–3d** : $R = CH_2(CH_2)_2CH_3$

1e–3e : $R = CH_2(CH_2)_3CH_3$ **1f–3f** : $R = (CH_3)_3C$ **1g–3g** : $R = C_6H_5CH_2$ **1h–3h** : $R = C_6H_5$

Scheme 1

2a–**2h** were prepared by the direct amination of 2-substituted 4,5-dichloropyridazinones with hydrazine hydrate in ethanol under microwave irradiation at 80 °C. The reaction times were decreased from more than 6 hours to 18 minutes and the yields were also improved a little compared to those obtained by the conventional heating method, which was reported previously [12]. The final compounds **3a**–**3h** were obtained by condensation of the 2-substituted 4-amino-3(2*H*)-pyridazinones with 2,6-difluorobenzoyl isocyanate in toluene at room temperature.

The lowest-field protons in the ^{1}H NMR spectrum appear at $\delta = 6.22$ and 7.52 ppm (which are assigned to the H-5 and H-6 of the pyidazinone ring of **2a**–**2h**) as a doublet with a coupling constant of 4.60 Hz, indicating *ortho* coupling, whereas the H-5 and H-6 of the pyidazinone ring of **3a**–**3h** appear at $\delta = 7.74$ and 7.81 ppm. These values are downfield due to the stronger electron-withdrawing benzoylurea.

Insecticidal Activities

We measured the biological activity of **3a**–**3h** against the armyworm, *Pseudaletia separata* Walker, according to the modified method described previously [11]. The bioassay tests show that some compounds exhibit moderate activity. The mortality of **3b**–**3f** was 72, 50, 43, 53, and 30 at 500 mg dm^{-3}, while **3a**, **3g**, and **3h** showed no activity. The length of the aliphatic chain at N-2 of the pyidazinone ring affect the activity. A shorter or longer carbon chain was unfavorable to activity, and a bulky group, such as phenyl, also decreased the activity.

In conclusion, we report a rapid, mild, and efficient procedure under microwave conditions for the synthesis of 2-substituted 4-amino-3(2*H*)-pyridazinones. The intermediates were successfully extended to synthesize some analogs of chitin synthesis inhibitors, **3a**–**3h**, attaching the 4-position of the 3(2*H*)-pyridazinone core to the benzoylurea moiety, which had been previously inaccessible by existing procedures. The easy preparation of 2-substituted 4-amino-3(2*H*)-pyridazinones should make it an ideal synthon for organic synthesis and potential application in biological research.

Experimental

All mp were obtained with an electrothermal digital apparatus made in Shanghai. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker WP-500SY spectrometer with *TMS* as internal standard. Chemical shifts are reported in δ (ppm) values. High-resolution mass spectra were recorded under electron

impact conditions using a MicroMass GCT CA 055 instrument. Infrared (IR) spectra were recorded in the range 4000–600 cm^{-1} using a Nicolet 470 infrared spectrometer. Microwave-promoted reaction was carried out on an Initiator (Biotage AB, Sweden). Analytical thin-layer chromatography (TLC) was carried out on precoated plates (silica gel 60 F254), and spots were visualized with UV light.

*Syntheses of 2-Alkyl-4-amino-3(2H)-pyridazinones **2a–2h***

A mixture of the dichloropyridazinone (**1**, 2.26 mmol), 1.13 g hydrazine hydrate (22.6 mmol), and 15 cm^3 ethanol was heated (microwave; 18 min, 85°C), then concentrated to dryness in *vacuo* and the residue was purified *via* column chromatography (elution with 1/4 ethyl acetate/petroleum ether) to give solids or oils.

2-Methyl-4-amino-3(2H)-pyridazinone (**2a**, $C_5H_7N_3O$)

Yield 58%; brown solid; mp 175–176°C; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 3.82$ (s, CH_3), 4.98 (s, NH_2), 6.24 (d, $J = 4.83$ Hz, PyH), 7.51 (d, $J = 4.76$ Hz, PyH) ppm; MS (70 eV): $m/z(\%) = 125$ (M^+, 42), 96 (22), 69 (72), 54 (100), 40 (36).

2-Ethyl-4-amino-3(2H)-pyridazinone (**2b**, $C_6H_9N_3O$)

Yield 64%; brown solid; mp 120–122°C; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 1.38$ (t, $J = 7.25$ Hz, CH_3), 4.22 (q, $J = 7.25$ Hz, CH_2N), 5.04 (s, NH_2), 6.23 (d, $J = 4.76$ Hz, PyH), 7.51 (d, $J = 4.76$ Hz, PyH) ppm; MS (70 eV): $m/z(\%) = 139$ (M^+, 96), 111 (100), 83 (30), 69 (46), 33 (28).

2-Propyl-4-amino-3(2H)-pyridazinone (**2c**, $C_7H_{11}N_3O$)

Yield 62%; oil; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 0.96$ (t, $J = 7.41$ Hz, CH_3), 1.85–1.89 (m, CH_2), 4.12 (t, $J = 7.36$ Hz, CH_2N), 5.02 (s, NH_2), 6.23 (d, $J = 4.80$ Hz, PyH), 7.53 (d, $J = 4.76$ Hz, PyH) ppm; MS (70 eV): $m/z(\%) = 153$ (M^+, 63), 125 (22), 111 (100), 83 (12).

2-n-Butyl-4-amino-3(2H)-pyridazinone (**2d**, $C_8H_{13}N_3O$)

Yield 68%; oil; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 0.95$ (t, $J = 7.34$ Hz, CH_3), 1.37–1.39 (m, CH_2), 1.76–1.82 (m, CH_2), 4.15 (t, $J = 7.41$ Hz, CH_2N), 5.03 (s, NH_2), 6.23 (d, $J = 4.71$ Hz, PyH), 7.53 (d, $J = 4.71$ Hz, PyH) ppm; MS (70 eV): $m/z(\%) = 167$ (M^+, 47), 139 (77), 111 (100), 83 (14), 69 (60).

2-Pentyl-4-amino-3(2H)-pyridazinone (**2e**, $C_9H_{15}N_3O$)

Yield 65%; oil; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 0.90$ (t, $J = 7.12$ Hz, CH_3), 1.32–1.38 (m, CH_2CH_2), 1.79–1.85 (m, CH_2), 4.19 (t, $J = 7.36$ Hz, CH_2N), 5.02 (s, NH_2), 6.22 (d, $J = 4.72$ Hz, PyH), 7.52 (d, $J = 4.72$ Hz, PyH) ppm; MS (70 eV): $m/z(\%) = 181$ (M^+, 76), 153 (63), 111 (100), 83 (12), 69 (46).

2-t-Butyl-4-amino-3(2H)-pyridazinone (**2f**, $C_8H_{13}N_3O$)

Yield 70%; solid; mp 109–111°C; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 1.69$ (s, $(CH_3)_3$), 5.29 (s, NH_2), 6.33 (d, $J = 4.72$ Hz, PyH), 7.59 (d, $J = 4.72$ Hz, PyH) ppm; MS (70 eV): $m/z(\%) = 167$ (M^+, 62), 111 (100), 83 (30), 57 (20), 55 (35), 41 (47).

2-Benzyl-4-amino-3(2H)-pyridazinone (**2g**, $C_{11}H_{15}N_3O$)

Yield 55%; brown solid; mp 90–91°C; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 4.99$ (s, NH_2), 5.22 (s, CH_2), 6.20 (d, $J = 4.78$ Hz, PyH), 7.25–7.42 (m, ArH), 7.53 (d, $J = 4.75$ Hz, PyH) ppm; MS (70 eV): $m/z(\%) = 201$ (M^+, 100), 91 (63), 69 (46).

2-Phenyl-4-amino-3(2H)-pyridazinone (**2h**, $C_{10}H_9N_3O$)

Yield 64%; solid; mp 135–136°C; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 4.55$ (s, NH_2), 6.27 (d, $J = 4.80$ Hz, PyH), 7.38–7.62 (m, ArH), 7.66 (d, $J = 4.80$ Hz, PyH) ppm; MS (70 eV): $m/z(\%) = 187$ (M^+, 100), 152 (21), 92 (20), 76 (30), 53 (32).

1-(2,6-Difluorobenzoyl)-3-(2-alkyl-3-oxopyridazin-4-yl)ureas ***3a–3h***

A solution of 0.48 g 2,6-difluorobenzoyl isocyanate (2.6 mmol) in 5 cm^3 dry toluene was added to a stirred solution of 2.5 mmol 2-alkyl-4-amino-3(2*H*)-pyridazinones **2a–2h** in 5 cm^3 dry toluene. The mixture was stirred overnight at room temperature, then the solvent was removed by evaporation under reduced pressure. The residue was chromatographed on a silica-gel column (petroleum ether (60–90°C) ethyl acetate 4/1 to 2/1) to afford the desired product.

1-(2,6-Difluorobenzoyl)-3-(2-methyl-3-oxopyridazin-4-yl]urea (**3a**, $C_{13}H_{10}F_2N_4O_3$)

Yield 58%; light yellow solid; mp 198–200°C; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 3.83$ (s, CH_3), 7.02–7.06 (m, ArH), 7.49–7.55 (m, ArH), 7.73 (d, $J = 4.68$ Hz, PyH), 7.86 (d, $J = 4.61$ Hz, PyH), 8.70 (s, NH), 11.37 (s, NH) ppm; IR (KBr): $\bar{\nu} = 3400$, 3120, 1730, 1710, 1650 cm^{-1}; MS (70 eV): $m/z(\%) = 308$ (M^+, 6), 165 (13), 141 (100), 113 (19); HRMS: calcd for $C_{13}H_{10}F_2N_4O_3$ 308.0720, found 308.0731; ^{13}C NMR (125 MHz, *DMSO*-d_6): $\delta = 39.7$, 111.9, 112.9, 113.2, 135.4, 137.7, 150.1, 155.5, 157.8, 159.8, 162.2 ppm.

1-(2,6-Difluorobenzoyl)-3-(2-ethyl-3-oxopyridazin-4-yl)urea (**3b**, $C_{14}H_{12}F_2N_4O_3$)

Yield 63%; light yellow solid; mp 217–218°C; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 1.39$ (t, $J = 7.12$ Hz, CH_3), 4.26 (q, $J = 7.02$ Hz, CH_2), 7.02–7.05 (m, ArH), 7.49–7.55 (m, ArH), 7.74 (d, $J = 4.59$ Hz, PyH), 7.78 (d, $J = 4.64$ Hz, PyH), 9.09 (s, NH), 11.38 (s, NH) ppm; IR (KBr): $\bar{\nu} = 3420$, 3220, 1750, 1700, 1660 cm^{-1}; MS (70 eV): $m/z(\%) = 322$ (M^+, 11), 165 (24), 141 (100), 137 (40), 113 (27); HRMS: calcd for $C_{14}H_{12}F_2N_4O_3$ 322.0877, found 322.0860; ^{13}C NMR (125 MHz, *DMSO*-d_6): $\delta = 13.4$, 46.9, 111.7, 112.9, 113.1, 135.5, 138.0, 150.0, 155.1, 157.8, 159.8, 162.2 ppm.

1-(2,6-Difluorobenzoyl)-3-(2-propyl-3-oxopyridazin-4-yl)urea (**3c**, $C_{15}H_{14}F_2N_4O_3$)

Yield 62%; white solid; mp 170–171°C; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 0.96$ (t, $J = 7.41$ Hz, CH_3), 1.82–1.88 (m, CH_2), 4.15 (q, $J = 7.34$ Hz, CH_2), 7.02–7.05 (m, ArH), 7.49–7.55 (m, ArH), 7.74 (d, $J = 4.69$ Hz, PyH), 7.81 (d, $J = 4.69$ Hz, PyH), 8.81 (s, NH), 11.38 (s, NH) ppm; IR (KBr): $\bar{\nu} = 3450$, 3180, 1750, 1710, 1680 cm^{-1}; MS (70 eV): $m/z(\%) = 336$ (M^+, 3), 157 (13), 141 (100), 113 (20); HRMS: calcd for $C_{15}H_{14}F_2N_4O_3$ 336.1034, found 336.1036; ^{13}C NMR (125 MHz, *DMSO*-d_6): $\delta = 10.9$, 21.3, 53.1, 111.7, 112.9, 113.1, 135.6, 137.8, 150.1, 155.3, 157.8, 159.8, 162.2 ppm.

1-(2,6-Difluorobenzoyl)-3-(2-n-butyl-3-oxopyridazin-4-yl)urea (**3d**, $C_{16}H_{16}F_2N_4O_3$)

Yield 57%; white solid; mp 217–218°C; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 0.89$ (t, $J = 7.33$ Hz, CH_3), 1.24–1.88 (m, CH_2), 1.67–1.73 (m, CH_2), 4.11 (q, $J = 7.08$ Hz, CH_2), 7.24–7.28 (m, ArH), 7.61–7.67 (m, ArH), 7.85 (d, $J = 4.56$ Hz, PyH), 7.91 (d, $J = 4.72$ Hz, PyH), 11.05 (s, NH), 11.84 (s, NH) ppm; IR (KBr): $\bar{\nu} = 3380$, 3130, 1720, 1700, 1680 cm^{-1}; MS (70 eV): $m/z(\%) = 350$ (M^+, 6), 271 (50), 165 (37), 141 (100), 113 (20); HRMS: calcd for $C_{16}H_{16}F_2N_4O_3$ 350.1190, found 350.1199; ^{13}C NMR (125 MHz, *DMSO*-d_6): $\delta = 13.6$, 19.3, 29.9, 51.2, 111.6, 112.7, 113.2, 135.8, 138.3, 150.3, 155.6, 157.4, 159.4, 162.8 ppm.

1-(2,6-Difluorobenzoyl)-3-(2-pentyl-3-oxopyridazin-4-yl)urea (**3e**, $C_{17}H_{18}F_2N_4O_3$)

Yield 66%; light yellow solid; mp 159–160°C; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 0.90$ (t, $J = 7.02$ Hz, CH_3), 1.32–1.38 (m, $2CH_2$), 1.79–1.85 (m, CH_2), 4.19 (q, $J = 7.41$ Hz, CH_2), 7.01–7.04 (m, ArH), 7.49–7.54 (m, ArH), 7.72 (d, $J = 4.77$ Hz, PyH), 7.75 (d, $J = 4.61$ Hz, PyH), 9.19 (s, NH), 11.40 (s, NH) ppm; IR (KBr): $\bar{\nu} = 3380$, 3210, 1730, 1710, 1660 cm^{-1}; MS (70 eV): $m/z(\%) = 364$ (M^+, 5), 179 (21), 157 (22), 141 (100), 113 (23); HRMS: calcd for $C_{17}H_{18}F_2N_4O_3$ 364.1347, found 364.1370; ^{13}C NMR (125 MHz, *DMSO*-d_6): $\delta = 13.8$, 21.7, 27.4, 28.2, 51.7, 111.7, 112.8, 113.5, 135.7, 137.9, 150.4, 155.8, 157.2, 160.1, 162.5 ppm.

1-(2,6-Difluorobenzoyl)-3-(2-t-butyl-3-oxopyridazin-4-yl)urea (**3f**, $C_{16}H_{16}F_2N_4O_3$)
Yield 71%; white solid; mp 221–222°C; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 0.97$ (s, $(CH_3)_3$), 7.02–7.05 (m, ArH), 7.49–7.55 (m, ArH), 7.68 (d, $J = 4.57$ Hz, PyH), 7.71 (d, $J = 4.51$ Hz, PyH), 9.13 (s, NH), 11.44 (s, NH) ppm; IR (KBr): $\bar{\nu} = 3410$, 3280, 1740, 1690, 1630 cm^{-1}; MS (70 eV): $m/z(\%) = 350$ (M^+, 30), 295 (66), 165 (37), 141 (100), 138 (83), 113 (60); HRMS: calcd for $C_{16}H_{16}F_2N_4O_3$ 350.1190, found 350.1185; ^{13}C NMR (125 MHz, *DMSO*-d_6): $\delta = 28.6$, 65.3, 111.6, 112.8, 113.5, 135.7, 137.5, 150.3, 155.6, 157.5, 159.6, 162.4 ppm.

1-(2,6-Difluorobenzoyl)-3-(2-benzyl-3-oxopyridazin-4-yl)urea (**3g**, $C_{19}H_{14}F_2N_4O_3$)
Yield 64%; light yellow solid; mp 210–211°C; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 5.35$ (s, CH_2), 6.99–7.02 (m, ArH), 7.26–7.34 (m, ArH), 7.43–7.51 (m, ArH and PyH), 7.72–7.74 (m, ArH and PyH), 9.10 (s, NH), 11.37 (s, NH) ppm; IR (KBr): $\bar{\nu} = 3410$, 3330, 1730, 1710, 1640 cm^{-1}; MS (70 eV): $m/z(\%) = 384$ (M^+, 17), 227 (46), 141 (98), 113 (20), 91 (100); HRMS: calcd for $C_{19}H_{14}F_2N_4O_3$ 384.1131, found 384.1124; ^{13}C NMR (125 MHz, *DMSO*-d_6): $\delta = 54.5$, 111.9, 113.2, 113.4, 122.4, 127.6, 127.9, 128.4, 135.7, 137.8, 150.2, 154.8, 157.5, 160.2, 162.4 ppm.

1-(2,6-Difluorobenzoyl)-3-(2-phenyl-3-oxopyridazin-4-yl)urea (**3h**, $C_{18}H_{12}F_2N_4O_3$)
Yield 63%; white solid; mp 198–199°C; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 7.02$–7.06 (m, ArH), 7.40–7.66 (m, ArH), 7.89 (d, $J = 4.66$ Hz, PyH), 7.93 (d, $J = 4.72$ Hz, PyH), 8.66 (s, NH), 11.51 (s, NH); IR (KBr): $\bar{\nu} = 3420$, 3310, 1720, 1680, 1640 cm^{-1}; MS (70 eV): $m/z(\%) = 370$ (M^+, 6), 213 (39), 157 (13), 141 (100), 113 (32); HRMS: calcd for $C_{18}H_{12}F_2N_4O_3$ 370.0956, found 370.0957; ^{13}C NMR (125 MHz, *DMSO*-d_6): $\delta = 111.9$, 113.2, 113.4, 125.7, 128.5, 128.8, 135.5, 138.0, 141.4, 150.0, 155.1, 157.8, 159.8, 162.2 ppm.

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