Copper Salt-Catalyzed Azide-[3 + 2] Cycloadditions of Ynamides and Bis-Ynamides

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Abstract: A one-pot synthesis of amide-substituted triazoles from alkyl bromides and ynamides is described here along with syntheses of novel bis-yn-

amides and their applications in [3+2] cycloadditions with azides to construct unique bis-triazoles.

Keywords: azide-[3 + 2] cycloaddition; bis-triazoles; bis-ynamides; Cu(I) catalysis; triazoles; ynamides

Introduction

The versatility of 1,3-dipolar cycloadditions^[1,2] in constructing synthetically and medicinally useful heterocycles^[3] has led us to investigate [3+2] cycloadditions of ynamides.^[4-8] Specifically, we have examined the classic Huisgen's azide-[3+2] cycloaddition^[9-11] given its current resurgence facilitated by the "click" chemistry concept.^[12] Recently, Cintrat^[13] and we^[14] independently communicated azide-[3 + 2] cycloadditions employing ynamides in a highly regioselective manner that favors 1,4-adducts $(1+2\rightarrow 3 \text{ in Figure 1})$. Particularly, we focused on developing tandem azidination- and hydroazidination-[3+2] cycloadditions of ynamides, leading to an array of amide-substituted triazoles.^[14] We report here details of our work with copper salt-catalyzed azide-[3+2] cycloadditions of vnamides that feature a one-pot synthesis of triazoles from alkyl bromides and constructions of bis-triazoles from novel bis-ynamides.



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Results and Discussions

Problems with the Competing Ynamide Hydrolysis

The feasibility of [3+2] cycloadditions of ynamides with organic azides could be readily established. As shown in Scheme 1, the CuSO₄·5 H₂O-catalyzed cycloaddition of terminal ynamide **5** with BnN₃ led to chiral amide-substituted triazole **7** in 82% yield as the



Scheme 1. Huisgen's azide-[3+2] cycloadditions of yn-amides.



only regioisomer using Fokin–Sharpless conditions.^[15] The catalytic sequence likely proceeds through a Cu(I) catalytic species generated via reduction of CuSO₄.^[15] The high level of regioselectivity is likely a result of the copper catalysis and steric factors because of the large oxazolidinone motif.^[15,16] However, there was an unresolved problem in our earlier work. When ynamides that are more prone to hydrolysis, such as **9**, were used the desired triazole **10** was obtained in only 30% yield with the hydrolysis product **11** being predominant.

To circumvent the hydrolysis problem, an improved protocol (Conditions B) was developed using 0.2 equivs. CuBr as the source of the Cu(I) catalyst along with anhydrous CH₃CN serving as the solvent and Et₃N as the base and ligand to improve the solubility of CuBr in CH₃CN. Under these new conditions, the yield of triazole 10 was improved to 56% without noticeable hydrolysis even when CH₃CN was not anhydrous, although the yield dropped to 44% (Scheme 1). The improvement was further visible in the reaction of ynamide 9 with azide 12, which led to the desired triazole 13 in 80% yield along with intriguingly 10% of the debrominated product 14, although 1.0 equiv. of CuBr was used. Under Conditions A, we could only observe a trace amount of 13 with hydrolysis of 9 being the major pathway.

Synthetic Scope of Organic Azides

To expand the synthetic scope, we embarked on an exploration of other organic azides besides BnN_3 . However, due to the difficulties in handling organic azides in general,^[17] especially those with low molecular weights, we ultimately designed a one-pot syntheses of amide-substituted triazoles from alkyl bromides directly. As shown in Scheme 2, organic azides were generated *in situ* by reacting alkyl bromides with NaN₃ in DMSO solution,^[18,19] and subsequently, copper salt, Na ascorbate, and terminal ynamides **5** and **15** were added without isolating the intermediate organic azides. Under this one-pot protocol, triazoles **17** and **18** were obtained in excellent yields from **5** and **15**, respectively.

The scope of this protocol was explored as shown in Table 1. In general, all primary bromides (en-



Scheme 2. One-pot synthesis of triazoles from alkyl bromides.

 Table 1. Scope of one-pot synthesis of triazoles from alkyl bromides.

entry	ynamides	R-Br ^a	1,4-cycloadducts	yie	eld [%] ^b
1	Ts N-== 19 Bn	BrOTBS 20	Ts_N ^{SN} ,OTB	S 27	70
2	19	Br0 21		28	78
3	19	Br 22		29	67
4	19	Br	Bn Br r	ОМе 30	10
5	19	Br 23	Ts	30	80 ^c
6	0 N 5	Br OBn 24	O ^{Bn} N ^{≥N} N−O N O ^N Ph.	Bn 31	73
7	Ph 5	Br 25		32	85
8	5	Br	O N ^{≥N} N N O Ph	33	40
9 10	0 N N Ph 9	Br OMe 23 Br 23	N≥N N Ph MeO	3r 13 13	10 50 ^c

^[a] 1.4 equivs. of NaN₃, 1.3 equivs. of R-Br, DMSO, room temperature, 12 h, and then, 1.0 equiv of ynamide, 0.10 equiv of CuSO₄·5 H_2O , 0.10 equiv of Na ascorbate, H_2O , room temperature, 14 h.

^[c] 1.4 equivs. of NaN₃, DMSO, room temperature, 12 h, 1.3 equivs. of R-Br, and then 1.0 equivs. of ynamide, 0.20 equivs. of CuBr, 2.0 equivs. of Et₃N, CH₃CN, room temperature, 12 h.

tries 1–3, 5–7 and 10) afforded the respective triazoles in good yields, including allyl bromide (entry 3), propargyl bromide (entry 7), and benzyl bromide (entries 5 and 10). A secondary bromide (entry 8) gave slightly lower yield. Many functional groups such as silyl ether (entry 1), benzyl ether (entry 5), acetal (entry 2), aryl bromide (entries 5 and 10), alkene (entry 3), and internal alkyne (entry 7) could be tolerated.

It is noteworthy that to avoid competing hydrolysis of ynamides during the cycloaddition, when using ynamide 9, the desired triazole 13 could be again obtained in 52% yield through the usage of new conditions [see Note c] related to Conditions B illustrated in Scheme 1 for the cycloaddition (entry 10). In addition, we found that sulfonyl-substituted ynamides such as 19 can also be problematic in terms of hydrolysis. Thus, when applicable, the cycloaddition of 19

^[b] Isolated yields.



Scheme 3. Fused triazoles employing macrocyclic ynamides.

with the azide derived from bromide **23** could also be improved to 80% (entry 4 versus 5).

Syntheses of Uniquely Fused Triazoles

In our previous communication,^[14] we had demonstrated that the Huisgen azide-[3 + 2] cycloaddition of internal ynamides was also feasible under thermal condition. As shown in Scheme 3, *n*-hexyl-substituted internal ynamide **34** could react with BnN₃ at 140°C in toluene to give 1,4-disubstituted triazole **35** in 58% yield with no observable 1,5-adduct **36**.



Scheme 4. One-pot syntheses of bistriazoles from dibromides.

Based on this precedent, we examined cycloadditions of macrocyclic ynamides, which could be prepared through a Cu(I)-catalyzed intramolecular coupling of amides tethered to alkynyl bromides that we had reported recently.^[20] As a result, novel macrocyclic fused triazoles **37** and **38** were successfully isolated in good yields when their respective macrocyclic ynamides were heated with BnN₃ at 140 °C in toluene in a sealed tube.

Novel Bis-Triazoles and Bis-Ynamides

With azide-[3+2] cycloadditions of ynamide firmly established, we examined two potential approaches for achieving double cycloadditions to construct novel bis-triazoles. Two bis-triazoles could be quickly assembled in one-pot with good yields by reacting ynamide **19** with di-azides generated *in situ* from dibromides **39a** and **39b** and NaN₃, thereby providing the feasibility of the first approach (Scheme 4).

To explore the scope of the bis-triazole synthesis, we achieved the synthesis of novel bis-ynamides **42a** and **42b** by coupling the respective dialkynyl bromides **41a** and **41b** with Evans auxiliaries in good yields (Scheme 5). Another approach to the bis-ynamide synthesis was the coupling of bis-amides with monoalkynyl bromide, which was demonstrated in Scheme 5. We successfully synthesized two terminal bis-ynamides **45** and **46** in reasonable overall yields through a two-step sequence: 1) coupling of TIPS substituted alkynyl bromide with bi-sulfonamide **43** or urethane **44**, and 2) the removal of the TIPS group using TBAF. Although bis-ynamides **42a**, **42b**, **45** and **46** are structurally novel, Diederich^[7r] had prepared some related ynamides.

With these bis-ynamides in hand, we examined the second approach to bis-triazoles (Scheme 6). Thermally driven [3 + 2] cycloadditions of internal bis-ynamides 42 a and 42b with BnN₃ succeeded in giving bis-triazoles 47a and 47b in moderate yields, while the



Scheme 5. Preparations of novel bis-ynamides.

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Scheme 6. Syntheses of bis-triazoles from bis-ynamides.

Fokin–Sharpless copper salt-catalyzed cycloadditions of terminal bis-ynamides **45** and **46** proceeded well to afford bis-triazoles **48** and **49**, respectively, in good yields.

Conclusions

We have described here a one-pot regioselective synthesis of amide-substituted triazoles from alkyl bromides and ynamides, and the synthesis of novel bisynamides and their applications to synthesize unique bis-triazoles.

Experimental Section

Triazole 7a with Conditions A

To a vial charged with ynamide 5 (50.0 mg, 0.267 mmol) added CuSO₄·5 H₂O (3.3 mg, 0.0134 mmol, were 0.050 equivs.), Na ascorbate (2.70 mg)0.0134 mmol, 0.050 equivs.), benzyl azide (37.0 μL, 0.294 mmol, 1.10 equivs.), and tert-butyl alcohol (0.5 mL). The mixture was stirred at room temperature for 2 min before H₂O (1 mL) was added. The reaction mixture was then vigorously stirred at room temperature for 12 h under nitrogen. After TLC showed that ynamide 5 was all consumed, the reaction mixture was diluted with H₂O (8 mL) and extracted with EtOAc $(3 \times 10 \text{ mL})$. The combined organic layers were washed with saturated aqueous NaCl and dried over Na₂SO₄. Removal of solvent under reduced pressure gave crude triazole 7a, which was purified via silica gel flash column chromatography with EtOAc/hexane as gradient eluent to yield the pure triazole 7a as a white solid; yield: 70.0 mg (82%).

Triazole 10 with Conditions B

To a vial charged with ynamide 9 (15.0 mg, 0.070 mmol) were added CuBr (1.8 mg, 0.0035 mmol, 0.20 equivs.), BnN_3 (13.0 µL. 0.098 mmol, 1.40 equivs.), 19.5 µL Et₂N (0.14 mmol, 14.1 mg, 2.0 equivs.) and CH₃CN (1 mL). The reaction mixture was then vigorously stirred at room temperature for 12 h under nitrogen. After TLC showed that ynamide 9 was all consumed, the reaction mixture was diluted with H₂O (4 mL) and extracted with EtOAc (3×5 mL). The combined organic layers were washed with saturated aqueous NaCl and dried over Na₂SO₄. Removal of solvent under reduced pressure gave crude triazole 10, which was purified via silica gel flash column chromatography with EtOAc/hexane as gradient eluent to yield the pure triazole 10 as a white solid; yield: 12.6 mg (56%).

General Procedure for Triazoles in Table 1

A stock solution of 0.5 M NaN₃ in DMSO was prepared by stirring at room temperature for 3 h. To a vial charged with 1.40 equivs. of 0.5 M NaN₃ in DMSO solution was added 1.30 equivs. of an alkyl bromide. After the mixture had been stirred for 12 h, and TLC showed that alkyl bromide was all consumed, H₂O (1/5 the total volume of DMSO used above) was added followed by 1.00 equiv. of the corresponding ynamide, 0.10 equiv. of CuSO₄·5 H₂O and 0.10 equiv. of sodium ascorbate. After TLC showed the starting ynamide was all consumed, the reaction mixture was poured into dilute aqueous NH₄OH, and then extracted by EtOAc ($3 \times$ equal volumes). The combined organic layers were washed with saturated aqueous NaCl and dried over Na₂SO₄. Removal of solvent under reduced pressure gave crude triazole, which was purified via silica gel column flash chromatography with EtOAc/hexane as gradient eluent.

Thermal Cycloadditions

To a sealed tube charged with an ynamide (1.00 equiv.) in toluene solution was added BnN_3 (1.30 equivs.). The reaction mixture was heated at 140 °C for 14 h. Removal of toluene under reduced pressure gave the crude triazole, which was purified *via* silica gel flash column chromatography with

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using EtOAc/hexane as gradient eluent to yield the pure triazole.

Charcterization data for triazoles and bis-triazoles prepared can be found in the Supporting Information.

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