Title: Synthesis of Dinitrogen-Fused Spiro Heterocycles via Organocatalytic 1,3-dipolar Cycloaddition of 2-Arylidene-1,3-indandiones and Azomethine Imines

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Synthesis of Dinitrogen-Fused Spiro Heterocycles via Organocatalytic 1,3-dipolar Cycloaddition of 2-Arylidene-1,3-indandiones and Azomethine Imines

Jindian Duan,[a] Jing Cheng,[a] Yuyu Cheng,[a] and Pengfei Li*[a]

Dedication ((optional))

Abstract: An efficient 1,3-dipolar cycloaddition of 2-arylidene-1,3-indandiones with azomethine imine has been developed to furnish spiro indane-1,3-dione-pyrazolidinones in generally good to high yields with excellent diastereoselectivities under mild conditions.

Introduction

Heterocycles are privileged structural units that are frequently encountered in biologically active natural products as well as in pharmaceuticals and agrochemicals.[1] In particular, dinitrogen-fused heterocycles are the core moieties in many biologically active compounds and have been widely investigated as herbicides, pesticides, and analogues of β-lactam antibiotics such as penicillin and cephalosporin.[2] Therefore, the practical and efficient methodologies for the synthesis of dinitrogen-fused heterocycles have attracted much attention.[3] Among various methods, the metal-catalyzed and organocatalytic 1,3-dipolar cycloaddition based on azomethine imines stand out, and have been extensively investigated.[4] Specially, using *N,N*-cyclic azomethine imine as synthon provides an efficient access to dinitrogen-fused heterocycles.[5-6] Notable, several catalyst-free 1,3-dipolar cycloaddition of *N,N*-cyclic azomethine imines could proceed smoothly to furnish dinitrogen-fused heterocycles.[7]

Recently readily accessible 2-arylidene-1,3-indandiones have been widely used for the synthesis of spiro-indane-1,3-dione skeletons,[8] which are important scaffolds in medicinal chemistry[9] and also serve as versatile building blocks for the construction of many bioactive natural products, drugs, and therapeutic leads.[10] Due to the biomedical importance of dinitrogen-fused heterocycles and spiro-indane-1,3-dione skeletons, combination of these two units in one molecular benefits diversity of structure and drug discovery. However, to our best knowledge, 1,3-dipolar cycloaddition reactions of 2-arylidene-1,3-indandiones with 1,3-dipoles have not been reported. We aim at developing an organocatalytic efficient 1,3-dipolar cycloaddition between 2-arylidene-1,3-indandiones and azomethine imines for the construction of complex polycycles containing two bioactive units of dinitrogen-fused heterocycle and spiro-indane-1,3-dione.

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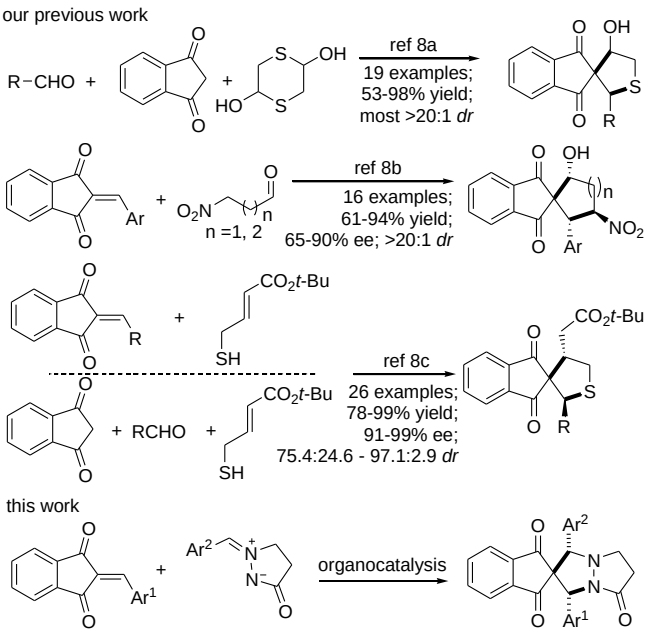
Based on our previous work and as a continuation of our research interests in synthesis of multicyclic spiro-1,3-indandiones,[8] here we disclose an organocatalytic catalyzed 1,3-dipolar cycloaddition of 2-arylidene-1,3-indandiones with *N,N*-cyclic azomethine imine to afford highly functionalized spiro indane-1,3-dione-pyrazolidinones in good yields with excellent diastereoselectivities under mild conditions (Scheme 1).



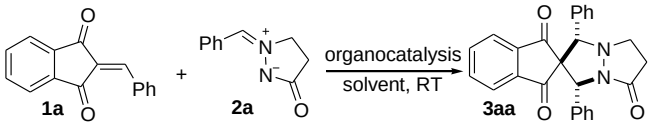
Scheme 1. Synthesis of spiro-indane-1,3-dione skeletons.

Results and Discussion

We initiated our investigation by evaluating the reaction of 2-benzylidene-1,3-indandione **1a** and azomethine imine **2a** in the presence of 5 mol % of Et_3N with CH_2Cl_2 as reaction media. To our gratification, the cycloaddition proceeded efficiently to provide the spiro indane-1,3-dione-pyrazolidinone **3aa** in 89% yield with excellent diastereoselectivity (>20:1dr) within 48 h (Table 1, entry 1). Encouraged by this promising result, several other bases were screened and Et_3N was found to be more suitable for the annulation (Table 1, entries 2-4). Subsequent investigations on the reaction media showed that 1,3-dipolar cycloaddition is highly dependent on the solvent used (Table 1, entries 5-9). It was found that the polar protic solvent MeOH was the most suitable solvent for this transformation in terms of both the reaction rate and the yield, affording the desired product in

98% yield with excellent diastereoselectivity (>20:1dr) in 48 h. Particularly, reducing the catalyst loading from 5 mol% to 1 mol%, spiro heterocycle **3aa** could be also obtained in 94% yield (Table, entry 10). Without catalyst, the 1,3-dipolar cycloaddition proceeded sluggishly to furnish **3aa** in 21% yield after 48 h but the diastereoselectivity kept at a high level (Table, entry 11).

Table 1. Optimization of Reaction Conditions.



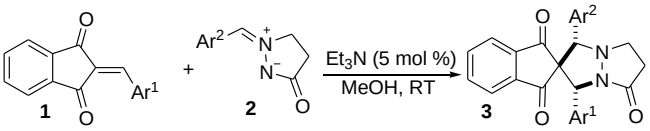
Entry[a]	Catalyst	Solvent	Time [h]	Yield [%][b]	dr[c]
1	Et_3N	CH_2Cl_2	48	89	> 20:1
2	DBU	CH_2Cl_2	48	70	> 20:1
3	DABCO	CH_2Cl_2	72	74	> 20:1
4	K_2CO_3	CH_2Cl_2	96	71	> 20:1
5	Et_3N	THF	72	72	> 20:1
6	Et_3N	MeCN	72	75	> 20:1
7	Et_3N	DMSO	48	Trace	> 20:1
8	Et_3N	EtOAc	96	86	> 20:1
9	Et_3N	MeOH	48	98	> 20:1
10[d]	Et_3N	MeOH	48	94	> 20:1
11[e]	-	MeOH	48	21	> 20:1

[a] Unless noted, **1a** (0.3 mmol), catalyst (5 mol %) and **2a** (0.36 mmol) in solvent (2 mL) was stirred at room temperature for the time given. [b] Yield of isolated product. [c] Determined by ^{1}H NMR spectroscopy of the crude mixture. [d] Et_3N (1 mol%) was used. [e] Et_3N (0 mol %) was employed.

Having established the optimum reaction conditions, a variety of 2-arylidene-1,3-indandiones **1** with azomethine imine **2** were tested to explore the reaction scope. As shown in Table 2, the 1,3-dipolar cycloaddition is considerably general and tolerates 2-arylidene-1,3-indandiones bearing either electron-rich or electron-deficient groups at the aromatic ring. Moreover, no significant electronic effect on the aromatic moiety of **1** was observed. It should be noted that all the reactions proceeded smoothly to furnish the expected products **3** in good to high yields (65-98%) with excellent diastereoselectivities (most products with 20:1 *dr*). 2-(4-(trifluoromethyl)benzylidene)-*2H*-indene-1,3-dione **1e** was found to react with **2a** to yield the product **3ea** with a diastereomeric ratio of 4:1 (Table 2, entry 5). And product **3ja** with a diastereomeric ratio of 7:1 was obtained from the cycloaddition between 2-(3-methoxybenzylidene)-2H-indene-1,3-dione **1j** and **2a** (Table 2, entry 10). It should be noted that thiophen-substituted 1,3-indandione **1k** readily participated in this reaction, giving rise to the desired product **3ka** in 70% yield with more than 20:1 *dr* (Table 2, entry 11).

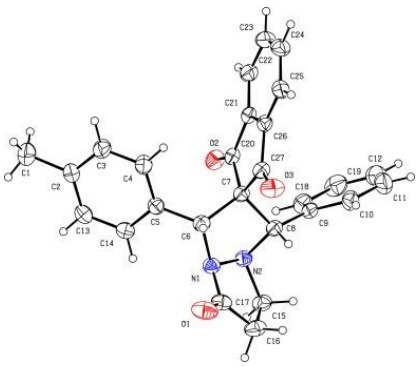
The scope was, however, restricted to azomethine imine **2a**. It was found that 1,3-dipolar cycloaddition between 2-benzylidene-1,3-indandione **1a** and azomethine imine with substituted-aromatic group **2** resulted in an intracable mixture of products no matter employing Et_3N as catalyst or catalyst-free conditions (Table 2, entries 12-13). 2-Alkylidene-1,3-indandione was also found to be incompatible with reaction system (Table 2, entry 14).

Table 2. Substrate Scope of 2-Arylidene-1,3-indandiones.



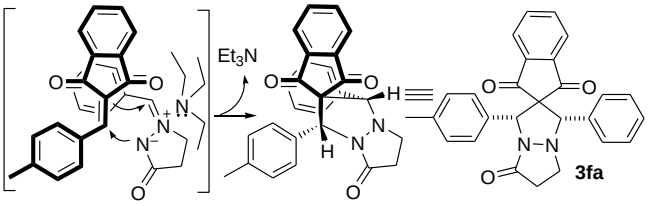
Entry[a]	Ar^1	Ar^2	Time [h]	Yield [%][b]	dr[c]
1	Ph	Ph	48	**3aa**, 98	> 20:1
2	4-FPh	Ph	48	**3ba**, 75	> 20:1
3	4-ClPh	Ph	48	**3ca**, 85	> 20:1
4	4-BrPh	Ph	48	**3da**, 84	> 20:1
5	4-CF_3Ph	Ph	48	**3ea**, 92	4:1
6	4-MePh	Ph	48	**3fa**, 83	> 20:1
7	3-BrPh	Ph	48	**3ga**, 65	> 20:1
8	3-NO_2Ph	Ph	72	**3ha**, 71	> 20:1
9	3-MePh	Ph	48	**3ia**, 93	> 20:1
10	3-MeOPh	Ph	48	**3ja**, 82	7:1
11	2-thienyl	Ph	72	**3ka**, 70	> 20:1
12	Ph	4-BrPh	72	-	-
13	Ph	4-MePh	72	-	-
14	i-Pr	Ph	72	-	-

[a] Unless noted, **1** (0.3 mmol), Et_3N (5 mol %) and **2a** (0.36 mmol) in MeOH (2 mL) was stirred at room temperature for the time given. [b] Yield of isolated product. [c] Determined by ^{1}H NMR spectroscopy of the crude mixture.



Scheme 2. X-ray structure of product **3fa**.

The structure of cyclization product **3fa** was unambiguously confirmed by single-crystal X-ray analysis (Scheme 2).[11] Two aromatic groups were found to be in the same side of the new formed five-membered ring. Based on the relative configuration of **3fa**, possible models for the intermediates in the 1,3-dipolar cycloaddition reaction were proposed. As depicted in Scheme 3, the cycloaddition was thought to be a concerted reaction. 2-(4-methylbenylidene)-1*H*-indene-1,3(2*H*)-dione **1f** interacted with azomethine imine **2a** via π-π stacking. Nitranion attacks less hindered, less substituted end of the double bond while more substituted end of the double bond reacts with carbon atom of imine ion to give the desired cycloaddition product **3fa**. Introducing substituent into benzene ring of azomethine imine **2** weakened the interaction between 2-arylidene-1,3-indandiones **1** and azomethine imine **2** as a result of increasing steric hindrance. This might be the reason that azomethine imine with substituted-aromatic ring could not be compatible in the transformation. Nevertheless, the exact catalytic mechanism of the cycloaddition still needs more investigation.



Scheme 3. Possible reaction models in [3 + 2] cycloaddition.

Conclusions

In summary, we have developed a Et_3N–catalyzed 1,3-dipolar cycloaddition of 2-arylidene-1,3-indandiones with *N,N*-cyclic azomethine imine to furnish spiro indane-1,3-dione-pyrazolidinones in generally good to high yields with excellent diastereoselectivities under mild conditions. This novel method tolerates a wide range of 2-arylidene-1,3-indandiones and is a reliable method for the rapid construction of valuable dinitrogen-fused heterocycles. Further investigations of the development of a catalytic asymmetric variant of this reaction and the application of this catalytic approach are currently underway in our laboratory.

Experimental Section

General Procedure for the synthesis of spiro indane-1,3-dione-pyrazolidinones **3**

To a stirred mixture of 2-arylidene-1,3-indandiones **1** (0.3 mmol) and azomethine imine **2** (0.36 mmol) in MeOH (2 mL) was added Et_3N (5 mol %) and then kept at room temperature for the time given, which was monitored by TLC. After removal of the solvent, the crude residue was purified by column chromatography (petroleum ether / ethyl acetate 3/1 v/v) on silica gel to give the corresponding products **3**.

NMR Data of a Representative Compound

1',3'-Diphenyl-6',7'-dihydro-1'H-spiro[indene-2,2'-pyrazolo[1,2-a]pyrazole] -1,3,5'(3'H)-trione (**3aa**): ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.97 (d, *J* = 7.6 Hz, 1H), 7.72–7.68 (m, 1H), 7.58–7.55 (m, 1H), 7.38 (d, J = 7.6 Hz, 1H), 7.19–7.18 (m, 2H), 7.16–7.06 (m, 8H), 5.81 (s, 1H), 4.42 (s, 1H), 3.85–3.79 (m, 1H), 3.10–3.02 (m, 2H), 2.94–2.87 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 198.3, 194.6, 172.5, 143.1, 141.3, 136.4, 135.6, 135.2, 131.6, 128.8, 128.5, 128.4, 127.8, 127.7, 125.7, 123.3, 123.2, 77.4, 72.1, 63.3, 48.2, 32.5.

Acknowledgements

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Keywords: organocatalysis • cycloaddition • 1,3-dipolar • 2-arylidene-1,3-indandione • azomethine imine

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[11] CCDC 1439843 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

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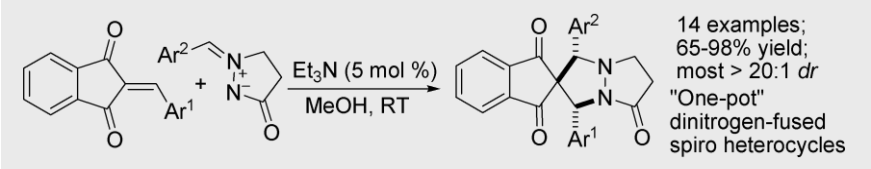

An efficient cycloaddition between 2-arylidene-1,3-indandiones and azomethine imines has been developed for the construction of dinitrogen-fused spiro heterocycles.

1,3-dipolar cycloaddition*

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*one or two words that highlight the emphasis of the paper or the field of the study