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Transition metal-free assembly of 1,3,5-triazines by using ethyl bromodifluoroacetate as C1 source

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An efficient transition metal-free annulation of amidine with ethyl bromodifluoroacetate to access 2,4-disubstituted-1,3,5-triazines is firstly presented. The desired symmetric and unsymmetric 2,4-disubstituted-1,3,5-triazines were obtained in decent yields via multiple C-N bonds formation, in which ethyl bromodifluoroacetate is harnessed as unique C1 synthon via quadruple cleavage. This reaction features with transition metal-free, oxidant-free and simple operation, in which only low toxic inorganic wastes were generated.

1,3,5-triazines are indisputably present as one of the privileged karyoskeletons in natural products and pharmaceutical molecules.[1] As showcased in Figure 1, a round of 1,3,5-triazines have been found with good biological activities.[2] Furthermore, 1,3,5-triazines could serve as promising ligands in organic synthesis for the assembly of organometallic materials, liquid crystals, and transition-metal catalysts.[3] Conventionally, 1,3,5-triazines are achieved through the cyclization reactions of amidines with a strand of formylating reagents such as diimino salt, *N*-[(dimethylamino) methylene]benzamidine, *N*-carbamoyl benzamidine, and α-methoxymethylene Meldrum's acid.[4] However, these known multi-step reactions suffer from the shortcomings such as harsh reaction conditions, narrow substrate scope and low yields. To address this drawback, myriads alternative synthetic strategies have been established for the construction of 1,3,5-triazines in the past few years.[5] A sequence of novel C1 synthons have been developed for cyclizations of amidines to assemble the 1,3,5-triazines by organic chemists. However, transition-metal catalysts and oxidants were obligatory to generate good yields of the desired 1,3,5-triazines for these newly discovered carbon synthons.[6] Given the characteristic biological activity and coordination ability of 1,3,5-triazines as well as the sustainable chemistry, the development of a metal-free and facile protocol to streamline synthesis of 1,3,5-triazines is still in demand.

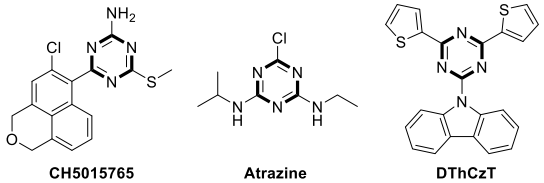
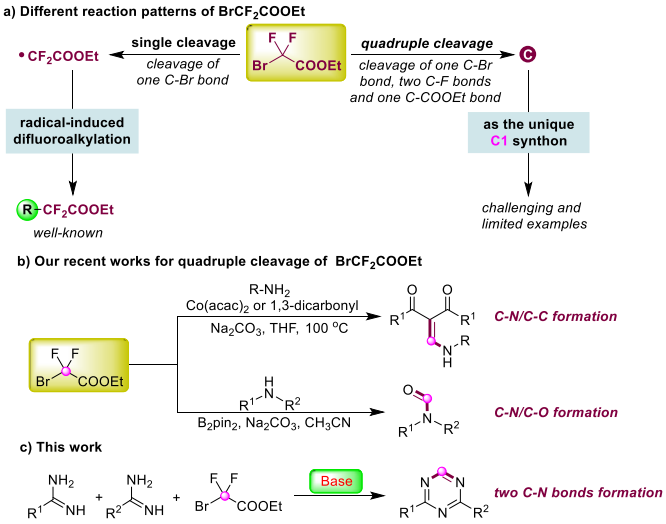


Figure 1. Representative examples of bioactive 1,3,5-triazines



Scheme 1. Novel methods using ethyl bromodifluoroacetate as a carbon source

Ethyl bromodifluoroacetate has been extensively employed as difluoroalkylating reagents in organic synthesis.[7,8] An ample number of radical-involved transformations have been developed using ethyl bromodifluoroacetate as radical precursors via single cleavage of C-Br bond.[7d-g,12] Despite of these undisputed difluoroalkylation of ethyl bromodifluoroacetate, the transition metal-free quadruple cleavage of ethyl bromodifluoroacetate is challenging and has been sparsely documented (Scheme 2a).[9,7b] In 2018, our group reported an efficient strategy for the assembly of β-aminoenones by cross-coupling of *in-situ* generated isocyanides, using ethyl bromodifluoroacetate as C1 source via

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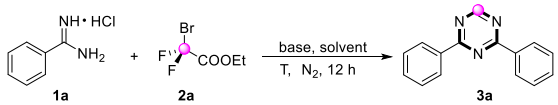
Table 1. Screening of optimized reaction conditions



1a 2a 3a

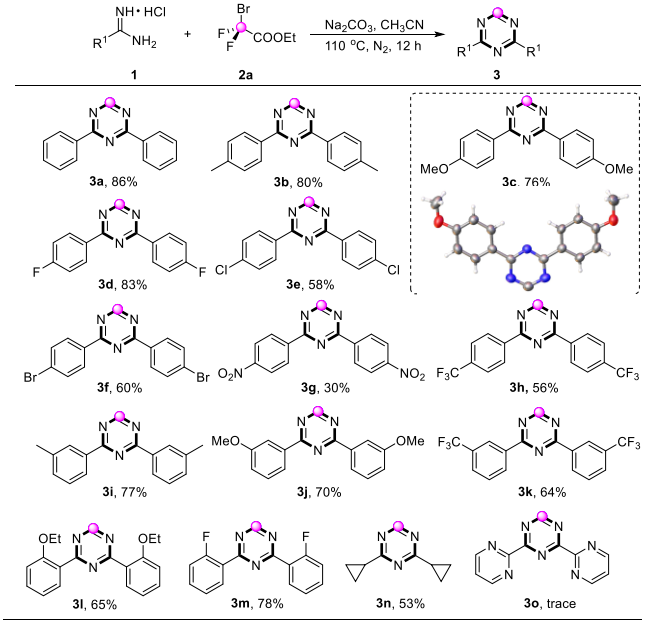
Entry[a]	Base	Solvent	Temperature (°C)	Yield (%)[b]
1	Na_2CO_3	CH_3CN	100	(78)[c]
2	K_2CO_3	CH_3CN	100	66 (57)
3	^{t}BuONa	CH_3CN	100	40
4	K_3PO_4	CH_3CN	100	50
5	MeOK	CH_3CN	100	35
6	Na_2CO_3	THF	100	22
7	Na_2CO_3	Acetone	100	45
8	Na_2CO_3	DCE	100	60
9	Na_2CO_3	DME	100	N.D.
10	Na_2CO_3	CH_3CN	80	(55)
11	Na_2CO_3	CH_3CN	110	(80)
12	Na_2CO_3	CH_3CN	120	(65)
13[d]	Na_2CO_3	CH_3CN	110	(63)
14[e]	Na_2CO_3	CH_3CN	110	(50)
15[f]	**Na_2CO_3**	**CH_3CN**	**110**	**(86)**
16[f,g]	Na_2CO_3	CH_3CN	110	(56)
17[f]	—	CH_3CN	110	<5

[a] Reaction conditions: **1a** (0.4 mmol), **2a** (3.0 equiv), base (4.0 equiv) in solvent (2 mL) for 12 h under N_2 atmosphere. [b] GC yields using dodecane as the internal standard. [c] isolared yields are given in parentheses. [d] 8 h. [e] 24 h. [f] **2a** (2.25 equiv). [g] Under air.

quadruple cleavage.[10] We also found that ethyl bromodifluoroacetate could be utilized as the formylation reagent, in which one C-N bond and one C-O bond were formed under transition metal-free conditions (Scheme 2b).[11] To our knowledge, taking advantage of ethyl bromodifluoroacetate as C1 synthon to construct multiple C-N bonds has yet to be explored. As our ongoing interest in transformation of $BrCF_2COOEt$,[12] herein, we describe a simple and green annulation of amidines to streamline synthesis of 1,3,5-triazines by using $BrCF_2COOEt$ as C1 synthon, which represents the first example for using $BrCF_2COOEt$ to forge the valuable 1,3,5-triazines under transition metal-free and oxidant-free conditions (Scheme 1c).

We commenced our study with benzamidine hydrochloride (**1a**) and ethyl bromodifluoroacetate (**2a**) as benchmark substrates and carried out the reaction in acetonitrile (CH_3CN) at 100 °C for 12 hours by using Na_2CO_3 as the base. To our delight and surprise, the 2,4-diphenyl-1,3,5-triazine **3a** was obtained in 78% yield (Table 1, entry 1). Inspired by this result, we subsequently inspected the effect of the different bases on this reaction (Table 1, entries 2-5). Among the bases investigated, Na_2CO_3 was proved to be the best choice, which exhibited higher reactivity than other bases, such as K_2CO_3, *t*BuONa, K_3PO_4 and MeOK. Successively, solvents screening for this transformation was executed (Table 1, entries 6-9). No desired product **3a** was observed when the reaction proceeded in ethylene glycol dimethyl ether. Meanwhile, when other solvents were used instead of CH_3CN, no superior results were gained. After determining the optimum base and solvent, the reaction temperature for this metal-free synthesis of 1,3,5-triazine was then studied (Table 1, entries 10-12). It was found that the isolated yield of **3a** was increased to 80% when reaction temperature was elevated to 110 °C (Table 1, entry 11). The attempt to shorten or extend the reaction time failed to deliver better results (Table 1, entries 13 and 14). Gladly, the isolated yield of **3a** was slightly increased to 86% when we reduced the amount of ethyl bromodifluoroacetate to 2.25 equivalents (Table 1, entry 15). The yield of 1,3,5-triazine **3a** was significantly decreased to 56% when the reaction was carried out under air atmosphere (Table 1, entry 16). Trace amount of targeted **3a** was detected when this cyclization reaction was performed without base (Table 1, entry 17), which indicated that base was indispensable for this transformation.

Having affirmed the optimized reaction conditions, we then investigated the functional group compatibility and substrate universality involved in this reaction. Firstly, a range of amidines were inspected to assemble various symmetrical 2,4-disubstituted-1,3,5-triazines, which was summarized in Scheme 2. Benzimidamides having electron-donating groups were proved to be good candidates in this transformation, enabling to produce the expected 2,4-diaryl-1,3,5-triazines **3b** and **3c** in 80% and 76% yield, respectively. The structure of **3c** was accurately confirmed by X-ray single crystal diffraction. Halo-substituted benzimidamides **1d**-**1f** were also amenable to this reaction, delivering the desired 1,3,5-triazines **3d**-**3f** in 58%-83% yields. Compared with the electron-donating groups substituted benzimidamides, the benzimidamides bearing electron-withdrawing groups such as (NO_2 and CF_3) demonstrated relatively low reactivity, which resulted in **3g** and **3h** in moderate yields. In addition to *para*-substituted benzimidamides, the *meta*- and *ortho*-substituted benzimidamides could work smoothly underlying the standard conditions as well, providing the corresponding 1,3,5-triazines **3i**-**3m** in 50%-77% yields. Aside from the aromatic amides, the submitting of cyclopropanecarboximidamide **1n** to this transformation was also successful, leading to the formation of 2,4-dicyclopropyl-1,3,5-triazine **3n** in 53% yield. Regrettably, only trace amounts of desired product **3o** was detected when



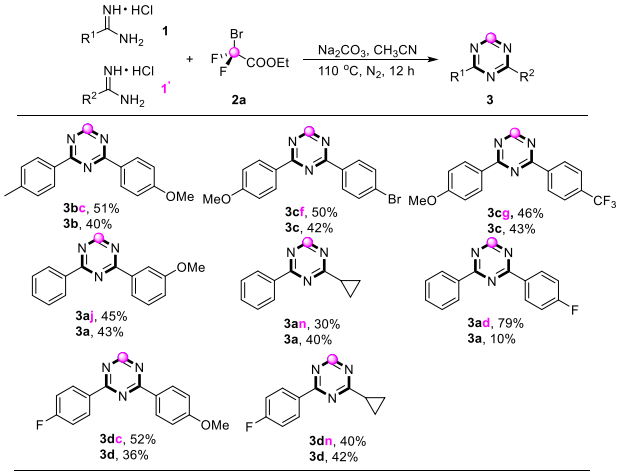
[a] Reaction conditions: **1a** (0.4 mmol), **2a** (2.25 equiv), Na_2CO_3 (4.0 equiv) in CH_3CN at 110 °C for 12 h under N_2 atmosphere.

Scheme 2. Synthesis of symmetrical 2,4-disubstituted-1,3,5-triazines

pyrimidine-2-carboximidamide **1o** was used as starting material.

Inspired by the success for the construction of symmetrical 2,4-disubstituted-1,3,5-triazines, we attempted to synthesize unsymmetric 2,4-disubstituted 1,3,5-triazines by utilizing two different amidines (Scheme 3). When the reaction of 4-methylbenzimidamide **1b** and 4-methoxybenzamidine **1c** was performed by using equimolar **1b** and **1c**, the yield of unsymmetrical product **3bc** was relatively low and symmetrical product **3b** was detected as the major product. To enhance the yield of unsymmetrical 2,4-disubstituted 1,3,5-triazines, the amount of two amidines was screened. Pleasurably, when the reaction was conducted with 2 equiv of **1b** and 1 equiv of **1c**, the yield of unsymmetric product **3bc** was increased to 51% along with 40% yield of **3b**. When **1b** was replaced with **1f** and **1g**, unsymmetric **3cf** and **3cg** were obtained with yields of 50% and 46%, respectively. Alternatively, the reaction of **1j** and **1n** with **1a** also delivered unsymmetric product **3aj**, **3an** in 45% and 30% yield. To our surprise and delight, the reaction of **1a** with 4-fluorobenzamidine **1d** afforded an unsymmetric product **3ad** with a high yield of 79%, with relatively low yield (10%) of symmetrical **3a**. The use of **1c** and **1n** instead of **1a** to react with **1d** was also successful, which enabled to furhish the unsymmetric 2,4-disubstituted 1,3,5-triazines **3dc** and **3dn** in yield of 52% and 40%, respectively.

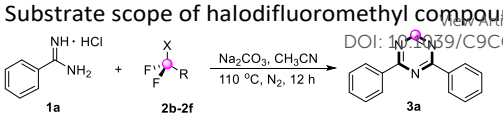
As summarized in Table 2, we also investigated other halodifluoromethyl compounds and it was found that all of them could serve as carbon sources to forge 2,4-substituted 1,3,5-triazine under the standard conditions by using benzamidine **1a** as cyclization reagent. In addition to ethyl bromodifluoroacetate **2a**, ethyl difluoroiodoacetate **2b** and ethyl difluorochloroacetate **2d** were proved to be good C1 source in this transformation, enabling to deliver **3a** in 75% and 77% respectively. When difluorobromoacetic acid **2c** was used as C1 source, the yield of **3a** could be isolated in 63% yield. Diethyl difluorobromomethylphosphonate **2e** and difluorobromomethyl morpholinone **2h** provided the corresponding 2,4-diphenyl 1,3,5-triazine **3a** in semblable yields of 55% and 54%, respectively. Compared with other

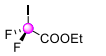
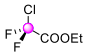
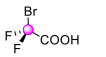
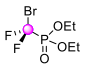
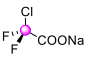
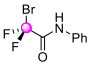
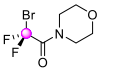


[a] Reaction conditions: **1** (0.6 mmol), **1'** (0.3 mmol), **2a** (3,0 equiv), Na_2CO_3 (4.0 equiv) in CH_3CN at 110 °C for 12 h under N_2 atmosphere. The isolated yields are given.

Scheme 3. Synthesis of unsymmetrical 2,4-diaryl-1,3,5-triazines

Table 2. Substrate scope of halodifluoromethyl compounds



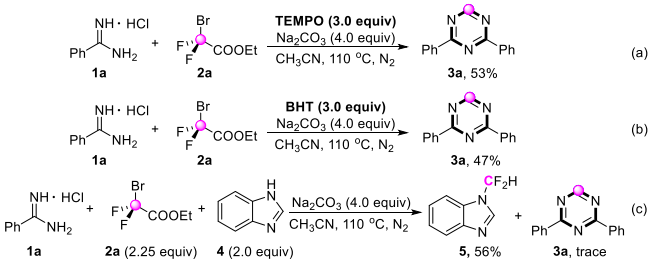
Entry	Difluorine reagents		Yield of **3a** (%)[a]
1		**2b**	75
2		**2c**	63
3		**2d**	77
4		**2e**	55
5		**2f**	30
6		**2g**	40
7		**2h**	54

Reaction conditions: **1a** (0.4 mmol), **2** (2.25 equiv), Na_2CO_3 (4.0 equiv), CH_3CN (2 mL) was stirred at 110 °C under N_2 for 12 h. [a] Isolated yield are given.

halodifluoromethyl compounds, sodium difluorochloroacetate **2f** and N-phenyl difluorobromoacetamide **2g** displayed lower reactivity as carbon sources, resulting in the target product **3a** with 30% and 40% yields, respectively.

To shed light on the reaction mechanism of this transformation, several control experiments were executed (Scheme 4). First, a radical-trapping experiment was conducted, the desired product **3a** was also isolated in moderate yields in the presence of 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) and 2,6-ditertbutyl-4-methylphenol (BHT) as the radical inhibitors, which indicated a radical pathway might not be involved in this transformation (Scheme 4a and 4b). Next, in order to capture the difluorocarbene, the carbene scavenger benzimidazole **4** was added to the reaction under standard conditions for 12 hours and N-difluoromethylbenzimidazole **5** was achieved in 56% isolated yield, along with trace amount of **3a** (Scheme 4c). Based on the above experimental results, it could be speculated that difluorocarbene (:CF_2) was actually generated *in-situ* during this transformation.

Based on our experimental results and previous work,[6,9] a plausible reaction mechanism for this synthesis of 1,3,5-triazine is depicted in Scheme 5. First, with the assistance of Base, $BrCF_2COOEt$ undergoes the saponification to produce $BrCF_2COONa$, which then suffers from the decarboxylation and debromination to generate difluorocarbenedifluorocarbene



Scheme 4. Control experiments for mechanistic studies

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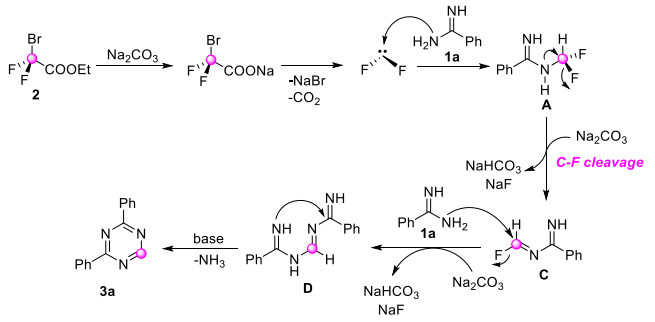

Scheme 5 Plausible mechanism

($:CF_2$) *in-situ*.[9] Successively, difluorocarbene is captured by amidine **1** to afford intermediate **A**, which is further transformed to imine intermediate **C** via one defluorination process under the basic conditions. Subsequently, the active intermediate **C** is attacked by another molecule amidine to deliver the intermediate **D**, which goes through intramolecular nucleophilic addition to furnish the targeted product **3a** by elimination of one molecule of ammonia.

In summary, we have developed a facile and efficient method for the synthesis of symmetric and unsymmetric 2,4-disubstituted-1,3,5-triazines under transition metal-free and external oxidant-free conditions with only low toxic inorganic wastes generated. The annulation of amidines with ethyl bromodifluoroacetate provided a range of 1,3,5-triazines in decent yields via multiple C-N bonds formation, in which quadruple cleavage of ethyl bromodifluoroacetate was achieved by cleavage of two C-F bonds, one C-Br bond and one C-COOEt bond in one-pot. This novel reactivity of ethyl bromodifluoroacetate for the assembly of 1,3,5-triazines is reported for the first time. Further studies for using ethyl bromodifluoroacetate as unique C1 synthon are underway in our laboratory.

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Conflicts of interest

The authors declare no competing financial interest.

Notes and references

1 (a) T. Irikura, Y. Abe, K. Okamura, K. Higo, A. Maeda, F. Morinaga, G. Shirai, S. Hatae, *J. Med. Chem.,* 1970, **13**, 1081; (b) Y. Iino, T. Karakida, N. Sugamata, T. Andoh, H. Takei, M. Takahashi, S. Yaguchi, T. Matsuno, M. Takehara, M. Sakato, S. Kawashima, Y. Morishita, *Anticancer Res*., 1998, **18**, 171; (c) B. Klenke, M. Stewart, M. P. Barrett, R. Brun, I. H. Gilbert, *J. Med. Chem.,* 2001, **44**, 3440; (d) R. V. Patel, P. Kumari, D. P. Rajani, K. H. Chikhalia, *Eur. J. Med. Chem.,* 2011, **46**, 4354.

2 (a) L. Whitesell, S. L. Lindquist, *Nat. Rev. Cancer.,* 2005, **5**, 761; (b) A. R. Katritzky, D. C. Oniciu, I. Ghiviriga, R. A. Barcock, *J. Chem. Soc., Perkin Trans.,* **2**, 1995, 785; (c) W. Zhu, Y. Liu, Y. Zhao, et. al. *Arch. Pharm. Chem. Life. Sci.,* 2012, **345**, 812; (d) Z.-F. An, R.-F. Chen, J. Yin, G.-H. Xie, H.-F. Shi, T. Tsuboi and W. Huang, *Chem. Eur. J.,* 2011, **17**, 10871.

3 (a) S. M. Gadekar, J. L. Frederick, *J. Org. Chem.,* 1962, **27**, 1383; (b) S. Naik, M. Kumaravel, J. T. Mague, M. S. Balakrishna, *Inorg. Chem.,* 2014, **53**, 1370; (c) C.-H. Lee, T. Yamamoto, *Bull. Chem. Soc. Jpn.,* 2002, **75**, 615; (d) M. Hernandez-Juarez, M. Vaquero, E. Alvarez, V. Salazar, A. Suarez, *Dalton. Trans.*, 2013, **42**, 351; (e) P. K. Santra, P. J. Sagar, *Mol. Catal. A: Chem.,* 2003, **197**, 37.

4 (a) H. Gold, *Angew. Chem.* 1960, **72**, 72956; (b) H. Bredereck, F. Effenberger, A. Hofmann, *Chem. Ber.,* 1963, **96**, 3265; (c) K. R. Huffman, F. C. Schaefer, *J. Org. Chem.,* 1963, **28**, 1812; (d) P. Wessig, J. Schwarz, *Monatsh. Chem.,* 1995, **126**, 99.

5 (a) A. L. Isfahani, M. B. Iraj, V. Mirkhani, M. Moghadam, S. Tangestaninejad and R. Kia, *Adv. Synth. Catal.,* 2013, **355**, 957; (b) M. Wang, Y. Meng, W. Wei, J. Wu, W. Yu and J. Chang, *Adv. Synth. Catal.,* 2018, **360**, 86; (c) A. DiazOrtiz, A. de la Hoz, A. Moreno, A. Sanchez-Migallon, G. Valiente, *Green Chem.*, 2002, **4**, 339; (d) C. Zhang, M.-T. Ban, K. Zhu, L.-Y. Zhang, Z.-Y. Luo, S.-N. Guo, D.-M. Cui and Y. Zhang, *Org. Lett.,* 2017, **19**, 3947; (e) Y. Yan, C. Cui, J. Wang, S. Li and Y. Liu, *Adv. Synth. Catal.,* 2019, **361**, 1.

6 (a) X. Xu, M. Zhang, H. Jiang, J. Zheng and Y. Li, *Org. Lett.,* 2014, **16**, 3540; (b) H. Huang, W. Guo, W. Wu, C.-J. Li and H. Jiang, *Org. Lett*., 2015, **17**, 2894; (c) Q. You, F. Wang, C. Wu, T. Shi, D. Min, H. Chen and W. Zhang, *Org. Biomol. Chem.,* 2015, **13**, 6723. (d) Y. Yan, Z. Li, H. Li, C. Cui, M. Shi and Y. Liu, *Org. Lett.,* **2017**, 19, 6228; (e) A. R. Tiwari, B.M. Bhanage, *Green Chem.* 2016, **18**, 144; (f) Y. Yan, Z. Li, C. Cui, H. Li, M. Shi and Y. Liu, *Org. Biomol. Chem.,* 2018, **16**, 2629.

7 (a) Z. Feng, Q.-Q. Min, Y.-L. Xiao, B. Zhang and X. Zhang, *Angew. Chem., Int. Ed.,* 2014, **53**, 1669; (b) Z. Feng, Q-Q. Min, and X. Zhang, *Org. Lett.*, 2016, **18**, 44; (c) W.-H. Guo, H.-Y. Zhao, Z.-J. Luo, S. Zhang and X. Zhang, *ACS Catal.,* 2019, **9**, 38; (d) J.-W. Gu, Q.-Q. Min, L.-C. Yu and X. Zhang, *Angew. Chem., Int. Ed.,* 2016, **55**, 12270; (e) X. Nie, C. Cheng, G. Zhu, *Angew. Chem. Int. Ed.,* 2017, **56**, 1898; (f) P. Xu, G. Wang, Y. Zhu, W. Li, Y. Cheng, S. Li and C. Zhu, *Angew. Chem., Int. Ed.,* 2016, **55**, 2939; (g) J. Xie, T. Zhang, F. Chen, N. Mehrkens, F. Rominger, M. Rudolph and A. S. K. Hashmi, *Angew. Chem.,Int. Ed.,* 2016, **55**, 2934; (h) Q. Qi, Q. Shen and L. Lu, *J. Am. Chem. Soc.,* 2012, **134**, 6548; (i) M. Zhang, J.-H. Lin and J.-C. Xiao, *Angew. Chem.,* 2019, **131**, 6140; (j) J. Liao, L. Fan, W. Guo, Z. Zhang, J. Li, C. Zhu, Y. Ren, W. Wu and H. Jiang, *Org. Lett.,* 2017, **19**, 1008; (k) J. Xu, Z. Kuang, Q. Song, *Chin. Chem. Lett.* 2017, **29**, 963; (l) Y. Zhou, Z. Xiong, J. Qiu, L. Kong and G. Zhu, *Org. Chem. Front.,* 2019, **6**, 1022; (m) F. Shen, P. Zhang, L. Lu and Q. Shen, *Org. Lett.*, 2017, **19**, 1032; (n)D. Zhu, Y. Gu, L. Lu and Q. Shen, *J. Am. Chem. Soc.,* 2015, **137**, 10547; (o) Q. Xie, C. Ni, R. Zhang, L. Li, J. Rong and J. Hu, *Angew. Chem., Int. Ed.,* 2017, **56**, 3206; (p) Q. Xie, Z. Zhu, L. Li, C. Ni, J. Hu, *Angew. Chem., Int. Ed.,* 2019**, 58**, 6405.

8 (a) Wei. Li, W. Xu, J. Xie, S. Yu and C. Zhu, *Chem. Soc. Rev.,* 2018, **47**, 654; (b) Z. Feng, Y.-L. Xiao and X. Zhang, *Acc. Chem. Res.,* 2018, **51**, 2264.

9 X. Ma, S. Mai, Y. Zhou, G.-J. Cheng and Q. Song, *Chem. Commun.,* 2018, **54**, 8960.

10 X. Ma, Y. Zhou, and Q. Song, *Org. Lett.,* 2018, **20**, 4777.

11 X. Ma, S. Deng and Q. Song, *Org. Chem. Front.,* 2018, **5**, 3505.

12 (a) M. Ke, Q. Feng, K. Yang and Q. Song, *Org. Chem. Front.*, 2016, **3**, 150; (b) M. Ke and Q. Song, *J. Org. Chem.,* 2016, **81**, 3654; (c) M. Ke and Q. Song, *Chem. Commun.,* 2017, **53**, 2222; (d) M. Ke and Q. Song, *Adv. Synth. Catal.,* 2017, **359**, 384; (e) W. Fu and Q. Song, *Org. Lett.,* 2018, **20**, 393; (f) W. Kong, C. Yu, H. An and Q. Song, *Org. Lett.,* 2018, **20**, 4975; (g) X. Ma, Q. Xuan, Q. Song, *Acta. Chimica. Sinica.,* 2018, **76**, 972.