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## ORIGINAL ARTICLE

# Bromo-catalyzed photo esterification of benzylsilanes with alcohols under aerobic conditions



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**Abstract** A photoinduced esterification reaction has been developed. In this reaction, benzylsilane shows very nice reactivity and selectivity, affording the corresponding esters as the only products. Under photo irradiation, the highly active bromo radical can be generated from the pre-catalyst  $\text{CBr}_4$ , which would promote the initial C—Si bond cleavage of benzylsilane via single electron transfer and the final transformation of acetal into ester via proton transfer. The existence of oxygen can not only oxidize the *in situ* generated benzyl radical into aldehyde intermediate, but also assist to regenerate bromo radical from bromine anion.

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## 1. Introduction

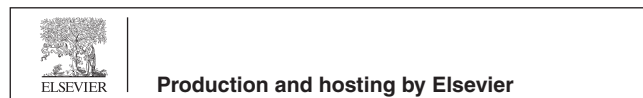
Esterification is a kind of important reaction which plays a key role in modern organic chemistry both in academia and industry [1–3]. Traditionally, esterification was commonly performed using carboxylic acids [4] or aldehydes [5] as starting materials to react with alcohols. Recently, the aerobic oxidative direct esterification of alcohols or aldehydes with alcohols using transition metal catalysts under mild conditions has

attracted great attention. Esters can also be prepared via the direct esterification of alcohols with silanes. Silicon chemistry has a rich history [6]. Owing to their unique properties and reactivities, transformations from silanes into various complex molecules are a widely used method in organic synthesis [7]. It is assumed that the esterification of alcohols with silanes can be beneficial, since the isolation of aldehyde from silanes can be avoided. Nevertheless, comparing to the well-established esterification of alcohols with carboxylic acids or aldehydes, direct esterification of alcohols with silanes are less developed [8]. Moran et. al. have reported iron(III) and nitrate ions mediated esterification of acylsilanes with alcohols, yielding the corresponding esters in moderate to excellent yields [9]. To the best of our knowledge, this is the only report regarding the direct esterification of alcohols with silanes. So far the photo-induced esterification of alcohols with silanes has not yet been reported. Photoinduced reactions have been receiving lot of attention in recent years [10], since such a transformation can be a constituent of the concept of green chemistry.

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To realize the above mentioned direct esterification, the highly efficient and selective break of the C—Si bond is very important. Recently, Guo et al. have reported the Br-catalyzed photo-cleavage of C—Si bond under aerobic conditions [11]. In such a photo-reaction, the bromo radical shows very nice catalytic activity in C—Si bond cleavage and the *in situ* generated aldehyde intermediates could be efficiently oxidized into benzoic acid derivatives yielding highly efficiently (Scheme 1). Based on this previous work, we hypothesized that one-pot synthesis from benzylsilanes into esters via aldehyde species under similar photo-oxidative conditions might be possible and did research in the field of photo-induced direct esterification of alcohols with silanes (Scheme 1). Herein, we would like to report our recent observation on the bromo-catalyzed photoinduced direct esterification of benzylsilanes with alcohols under aerobic conditions.

## 2. Experimental

### 2.1. General consideration

$^1\text{H}$  (400 MHz) NMR spectra of samples in  $\text{CDCl}_3$  (unless stated otherwise) were recorded on an AVANCE III 400 spectrometer.

### 2.2. General procedure for the photoreaction

Benzyltrimethylsilanes (0.20 mmol),  $\text{CBr}_4$  (0.06 mmol) and alcohols (5 mL) were added to a dry Pyrex reaction flask which was equipped with a magnetic stirrer. The mixture was irradiated by a Xe lamp (300 W) at rt under oxygen atmosphere. After completion of the reaction (monitored by TLC, eluent: petroleum ether:ethyl acetate = 10:1), the solvent was removed and the residue was purified by flash chromatography on silica gel (eluent: petroleum ether:ethyl acetate = 10:1) to afford the corresponding products.

*Methyl 4-methoxybenzoate (3aa)* [12]  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.99 (d,  $J$  = 8.8 Hz, 2 H), 6.91 (d,  $J$  = 8.4 Hz, 2 H), 3.88 (s, 3 H), 3.85 (s, 3 H).

*Methyl 4-ethoxybenzoate (3ba)* [13]  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.98 (d,  $J$  = 8.4 Hz, 2 H), 6.89 (d,  $J$  = 8.4 Hz, 2 H), 4.08 (q,  $J$  = 6.8 Hz, 2 H), 3.88 (s, 3 H), 1.34 (t,  $J$  = 6.9 Hz, 3 H).

*Methyl 4-(tert-butyl)benzoate (3ca)* [14]  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.97 (d,  $J$  = 8.4 Hz, 2 H), 7.45 (d,  $J$  = 8.4 Hz, 2 H), 3.90 (s, 3 H), 1.34 (s, 9 H).

*Methyl 4-phenylbenzoate (3da)* [15]  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.10 (d,  $J$  = 8.8 Hz, 2 H), 7.68–7.58 (m, 4 H), 7.48–7.42 (m, 2H), 7.41–7.35 (m, 1H), 3.93 (s, 3 H).

*Methyl benzoate (3ea)* [12]  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.06–8.02 (m, 2 H), 7.58–7.51 (m, 1 H), 7.46–7.42 (m, 2 H), 3.92 (s, 3 H).

*Methyl 4-fluorobenzoate (3fa)* [16]  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.05 (dd,  $J$  = 8.8, 5.2 Hz, 2 H), 7.11 (dd,  $J$  = 8.8, 5.2 Hz, 2 H), 3.91 (s, 3 H).

*Methyl 4-chlorobenzoate (3ga)* [12]  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.98 (d,  $J$  = 8.4 Hz, 2 H), 7.41 (d,  $J$  = 8.4 Hz, 2 H), 3.92 (s, 3 H).

*Methyl 4-bromobenzoate (3ha)* [12]  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.90 (d,  $J$  = 8.4 Hz, 2 H), 7.58 (d,  $J$  = 8.4 Hz, 2 H), 3.91 (s, 3 H).

*Methyl 4-(trifluoromethyl)benzoate (3ia)* [17]  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.16 (d,  $J$  = 8.0 Hz, 2 H), 7.71 (d,  $J$  = 8.4 Hz, 2 H), 3.96 (s, 3 H).

*Methyl 1-naphthoate (3ja)* [14]  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.91 (d,  $J$  = 8.8 Hz, 1 H), 8.17 (d,  $J$  = 7.2 Hz, 1 H), 8.00 (d,  $J$  = 8.0 Hz, 1 H), 7.87 (d,  $J$  = 8.0 Hz, 1 H), 7.87 (d,  $J$  = 8.0 Hz, 1 H), 7.64–7.58 (m, 1 H), 7.56–7.45 (m, 2 H), 4.00 (s, 3 H).

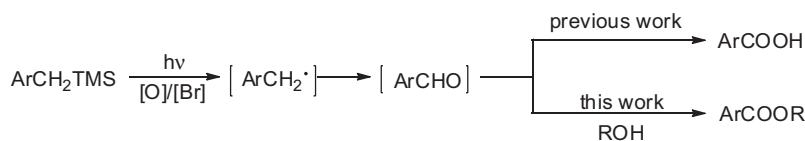
*Methyl 2-naphthoate (3ka)* [12]  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.60 (s, 1 H), 8.08–8.02 (m, 1 H), 7.93 (d,  $J$  = 8.0 Hz, 1 H), 7.87 (d,  $J$  = 8.8 Hz, 2 H), 7.60–7.50 (m, 2 H), 3.97 (s, 3 H).

*Ethyl 4-methoxybenzoate (3ab)* [18]  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.00 (d,  $J$  = 8.8 Hz, 2 H), 6.91 (d,  $J$  = 8.8 Hz, 2 H), 4.35 (q,  $J$  = 7.2 Hz, 2 H), 3.86 (s, 3 H) 1.38 (t,  $J$  = 7.2 Hz, 3 H).

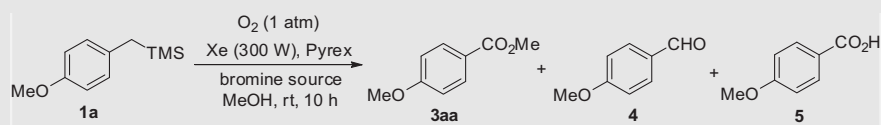
*Propyl 4-methoxybenzoate (3ac)* [19]  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.01 (d,  $J$  = 8.8 Hz, 2 H), 6.92 (d,  $J$  = 8.8 Hz, 2 H), 4.25 (t,  $J$  = 6.4 Hz, 2 H), 3.86 (s, 3 H) 1.78 (m, 2 H), 1.02 (t,  $J$  = 7.2 Hz, 3 H).

## 3. Results and discussion

To explore the feasibility of this reaction, (4-methoxybenzyl)trimethylsilane **1a** and methanol **2a** were chosen as the model substrates for the following studies. Firstly, HBr (aq. 40%) was used as the pre-catalyst under photo-oxidative conditions. It is satisfactory to see that the side reaction-oxidation of aldehyde into carboxylic acid was totally inhibited. However, only 3% of the desired ester was formed (entry 1, Table 1). To improve the yield of ester, other inorganic bromides were tested. Disappointingly, no desired product was afforded at all (entries 2–4, Table 1). Next, organic bromine sources NBS and  $\text{CBr}_4$  were applied in the reaction system. To our delight, 85% and 95% of the corresponding esters could be obtained, respectively (entries 5 and 6, Table 1).  $\text{CBr}_4$  was then chosen as the pre-catalyst for further studies. When reducing the amount of  $\text{CBr}_4$  from 30 to 10 mol%, both the yield and selectivity were decreased however (entries 7 and 8, Table 1). Without the introduction of pre-catalyst, no ester was afforded (entry 9, Table 1). The control experiments indicated that light (entry 10, Table 1) and oxygen (entry 11, Table 1) were also necessary for this reaction. Finally, the reaction was conducted under air atmosphere, affording **3aa** in 78% yield (entry 12,



**Scheme 1** Transformations of arylsilanes under photo-oxidative condition.

**Table 1** Optimization of reaction conditions.<sup>a</sup>

Entry	Bromine source (mol%)	NMR Yield <sup>b</sup> (%)		
		3aa	4	5
1	HBr (aq. 40%) (30)	3	20	0
2	LiBr·H <sub>2</sub> O + H <sub>2</sub> O (30)	0	40	0
3	NaBr + H <sub>2</sub> O (30)	0	15	0
4	KBr + H <sub>2</sub> O (30)	0	20	0
5	NBS (30)	85	0	0
6	CBr <sub>4</sub> (30)	95 (90) <sup>c</sup>	0	0
7	CBr <sub>4</sub> (20)	85	0	0
8	CBr <sub>4</sub> (10)	74	9	4
9	–	0	52	6
10 <sup>d</sup>	CBr <sub>4</sub> (30)	NR		
11 <sup>e</sup>	CBr <sub>4</sub> (30)	0 <sup>f</sup>		
12 <sup>g</sup>	CBr <sub>4</sub> (30)	78	1	3

<sup>a</sup> A solution of **1a** (0.2 mmol) and bromine source in anhydrous MeOH (5 mL) in Pyrex reaction tube was irradiated by a 300 W Xe lamp under oxygen atmosphere (1 atm) at rt for 10 h.

<sup>b</sup> Yield based on <sup>1</sup>H NMR analysis of the crude reaction mixture using CH<sub>2</sub>Br<sub>2</sub> as the internal standard.

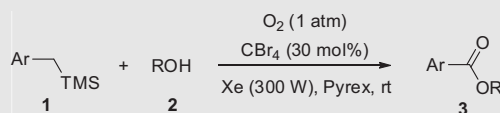
<sup>c</sup> Isolated yield.

<sup>d</sup> The reaction was carried without light.

<sup>e</sup> The reaction was carried out under argon atmosphere (1 atm).

<sup>f</sup> 56% of **1a** was recovered.

<sup>g</sup> The reaction was carried out under air atmosphere.

**Table 2** Photo esterification of benzyloxy silanes with alcohols under Condition A.<sup>a</sup>

Entry	1 (Ar)	2 (R)	3	Time (h)	Isolated yield (%)
1	<b>1a</b> (4-MeOC <sub>6</sub> H <sub>4</sub> )	<b>2a</b> (Me)	<b>3aa</b>	10	90
2	<b>1b</b> (4-EtOC <sub>6</sub> H <sub>4</sub> )	<b>2a</b> (Me)	<b>3ba</b>	10	91
3	<b>1c</b> (4- <sup>t</sup> BuC <sub>6</sub> H <sub>4</sub> )	<b>2a</b> (Me)	<b>3ca</b>	10	95
4	<b>1d</b> (4-PhC <sub>6</sub> H <sub>4</sub> )	<b>2a</b> (Me)	<b>3da</b>	10	97
5	<b>1e</b> (C <sub>6</sub> H <sub>5</sub> )	<b>2a</b> (Me)	<b>3ea</b>	10	88
6	<b>1f</b> (4-FC <sub>6</sub> H <sub>4</sub> )	<b>2a</b> (Me)	<b>3fa</b>	10	50
7	<b>1g</b> (4-ClC <sub>6</sub> H <sub>4</sub> )	<b>2a</b> (Me)	<b>3ga</b>	10	73
8	<b>1h</b> (4-BrC <sub>6</sub> H <sub>4</sub> )	<b>2a</b> (Me)	<b>3ha</b>	10	66
9	<b>1i</b> (4-CF <sub>3</sub> C <sub>6</sub> H <sub>4</sub> )	<b>2a</b> (Me)	<b>3ia</b>	24	26 (29) <sup>b</sup>
10	<b>1j</b> (1-naphthyl)	<b>2a</b> (Me)	<b>3ja</b>	10	75
11	<b>1k</b> (2-naphthyl)	<b>2a</b> (Me)	<b>3ka</b>	10	90
12	<b>1a</b> (4-MeOC <sub>6</sub> H <sub>4</sub> )	<b>2b</b> (Et)	<b>3ab</b>	20	77
13	<b>1a</b> (4-MeOC <sub>6</sub> H <sub>4</sub> )	<b>2c</b> ( <sup>i</sup> Pr)	<b>3ac</b>	15	67

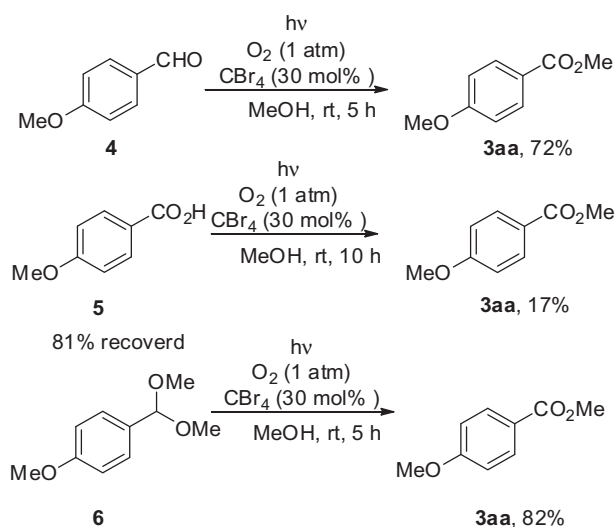
<sup>a</sup> A solution of **1** (0.2 mmol) and CBr<sub>4</sub> (30 mol%) in anhydrous ROH (5 mL) in Pyrex reaction tube was irradiated by a 300 W Xe lamp under oxygen atmosphere (1 atm) at rt.

<sup>b</sup> Recovered yield of **1i**.

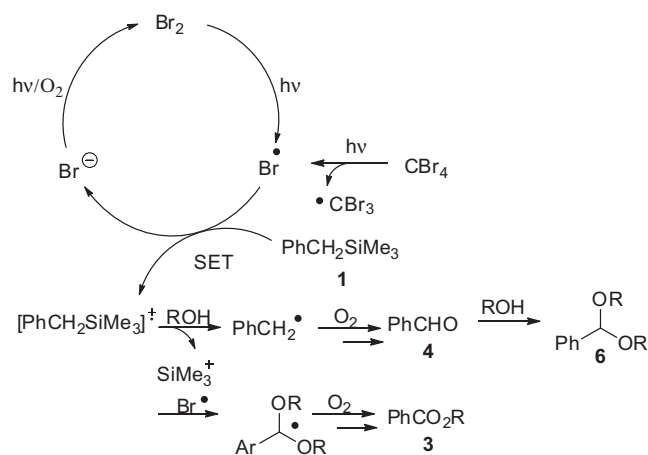
**Table 1**). Thus, Condition A (30 mol%  $\text{CBr}_4$ , Xe lamp (300 W), Pyrex,  $\text{O}_2$  (1 atm), and rt) was finally chosen as the standard condition for this photo esterification of benzylsilanes with alcohols.

With the optimal conditions in hand, we investigated the substrate scope of this photo esterification reaction under Condition A. Firstly, the electronic effect on the aryl rings was carefully studied (**Table 2**). Both strong and weak electron-donating silane substrates resulted in excellent ester yields (entries 1–4, **Table 2**). However, substrates bearing weak electron-withdrawing groups showed dramatic decrease in reactivity, in which only moderate yields could be achieved (entries 6–8, **Table 2**). When strong electron-withdrawing group like trifluoromethyl group was introduced in the substrate, the reaction gave even lower yield (entry 9, **Table 2**) with recovered starting materials. Naphthalene ring derived silanes were also tested, giving the corresponding esters also in excellent to moderate yields (entries 10 and 11, **Table 2**). The reaction proved to be also effective for the esterification of (4-methoxybenzyl)trimethylsilane **1a** with other alcohols such as ethanol and *n*-propanol (entries 12 and 13, **Table 2**).

To get some insights into the reaction mechanism, a set of control experiments were carried out (**Scheme 2**). According to our previous study [11], the photo oxidation of benzylsilanes **1** firstly leads to the corresponding aldehyde intermediate rather than the acid. We wonder whether the aldehyde is the intermediate for the examined esterification reaction. Thus, the photo esterification reaction of *p*-methoxybenzaldehyde **4** with methanol was then carried out under Condition A. The reaction could smoothly occur under condition A, with 72% of the corresponding methyl ester achieved. However, the esterification of *p*-methoxybenzoic acid **5** with methanol under Condition A only resulted in 17% of ester yield (with 81% of starting material recovered). These results indicated that the photo esterification should proceed via aldehyde intermediate but not the acid. The aldehyde intermediate may then further react with alcohol to form an acetal intermediate, which might be the key intermediate of the reaction. To further prove our assumption, the esterification of *p*-methoxybenzaldehyde dimethylacetal **6** with methanol was studied under Conditions A. It is satisfactory to note that 82% of ester could be



**Scheme 2** Study of reaction mechanism.



**Scheme 3** Plausible mechanism.

obtained. Thus, it can be deduced that aldehyde dimethylacetal should be the intermediate for this transformation.

Based on the experimental results and literature precedents [11,14,20–22], a photoinduced bromo-catalyzed esterification reaction of benzylsilanes and alcohols under aerobic conditions is proposed as shown in **Scheme 3**. Under photo irradiation, bromo radical is generated from the pre-catalyst  $\text{CBr}_4$ . This highly oxidative bromo radical intermediate may quickly transfer into bromine anion via single electron transfer (SET) from benzylsilane **1**. Meanwhile **1** converted into its radical cation, in which the C–Si bond has been greatly weakened. The radical cation may further undergo C–Si bond cleavage to form the benzyl radical in the presence of ROH, which can be easily oxidized into aldehyde **4** under aerobic condition. Next, **4** reacts with alcohol **2** to form acetal **6**. The subsequent proton transfer (PT) from **6** to bromo radical leads to HBr and radical species, which can be oxidized into ester **3** under aerobic photo-oxidative condition. Finally, bromo radical is regenerated from photooxidation of bromine anion by oxygen.

#### 4. Conclusion

In conclusion, we developed a photo-induced esterification employing benzylsilanes and  $\text{CBr}_4$  as catalyst. The benzylsilane derivatives showed good reactivity and selectivity, affording the corresponding esters as the only product. Mechanism studies suggest that the reaction undergoes a photo-induced SET from silane to bromo radical yielding a C–Si bond weakened silane radical cation, followed by the photooxidation of silane radical cation to aldehyde and subsequent to acetal intermediate, the proton transfer from acetal to bromo radical with the formation of acetal radical, and finally the photooxidation of acetal radical to ester product.

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**Appendix A. Supplementary data**

Supplementary data associated with this article can be found, in the online version, at <http://dx.doi.org/10.1016/j.jscs.2016.10.001>.

**References**

- [1] (a) D.J. Hart, D.C. Ha, *Chem. Rev.* 89 (1989) 1447–1465;  
(b) A. Das, P. Theato, *Chem. Rev.* 116 (2016) 1434–1495;  
(c) J.T. Stivers, R. Nagarajan, *Chem. Rev.* 106 (2006) 3443–3467.
- [2] (a) P.K. Mishra, D.G. Drueckhammer, *Chem. Rev.* 100 (2000) 3283–3310;  
(b) M.T. Dang, L. Hirsch, G. Wantz, J.D. Wuest, *Chem. Rev.* 113 (2013) 3734–3765.
- [3] (a) J.K. Puri, R. Singh, V.K. Chahal, *Chem. Soc. Rev.* 40 (2011) 1791–1840;  
(b) L.W. Xu, L. Li, G.Q. Lai, J.X. Jiang, *Chem. Soc. Rev.* 40 (2011) 1777–1790;  
(c) P.C. Bulman Page, S.S. Klair, S. Rosenthal, *Chem. Soc. Rev.* 19 (1990) 147–195.
- [4] H.J. Zhang, D.L. Priebbenow, C. Bolm, *Chem. Soc. Rev.* 42 (2013) 8450–8571.
- [5] J. Yoshida, K. Kataoka, R. Horcajada, A. Nagaki, *Chem. Rev.* 108 (2008) 2265–2299.
- [6] S. Farkas, *CNS Drug Rev.* 12 (2006) 218–235.
- [7] (a) H.F. Sore, W.R.J.D. Galloway, D.R. Spring, *Chem. Soc. Rev.* 41 (2012) 1845–1866;  
(b) Y. Nakao, T. Hiyama, *Chem. Soc. Rev.* 40 (2011) 4893–4901.
- [8] A. Hosomi, *Acc. Chem. Res.* 21 (1988) 200–206.
- [9] A.F. Patrocínio, P.J.S. Moran, *Synthetic Commun.* 30 (2000) 1419–1423.
- [10] (a) C.K. Prier, D.A. Rankic, D.W.C. Macmillan, *Chem. Rev.* 113 (2013) 5322–5353;  
(b) D.M. Schultz, T.P. Yoon, *Science* 343 (2014) 985;  
(c) P. Wang, M. Cheng, Z. Zhang, *J. Saudi Chem. Soc.* 18 (2014) 308–316;  
(d) A.B. Lavand, Y.S. Malghe, *J. Saudi Chem. Soc.* 19 (2015) 471–478;  
(e) A. Ding, Y. Wang, R. Rios, J. Sun, H. Li, H. Guo, *J. Saudi Chem. Soc.* 19 (2015) 706–709.
- [11] J. Sun, Y. Wang, L. Han, D. Xu, Y. Chen, X. Peng, H. Guo, *Org. Chem. Front.* 1 (2014) 1201–1204.
- [12] K.K. Rajbongshi, M.J. Sarma, P. Phukan, *Tetrahedron Lett.* 55 (2014) 5358–5360.
- [13] P.C. Andrews, T. Beck, B.H. Fraser, P.C. Junk, M. Massi, B. Moubaraki, K.S. Murray, M. Silberstein, *Polyhedron* 28 (2009) 2123–2130.
- [14] S. Hirashima, T. Nobuta, N. Tada, T. Miura, A. Itoh, *Org. Lett.* 12 (2010) 3645–3647.
- [15] L. Ackermann, C.J. Gschrei, A. Althammer, M. Riederer, *Chem. Commun.* (2006) 1419–1421.
- [16] M. Irfan, T.N. Glasnov, C.O. Kappe, *Org. Lett.* 13 (2011) 984–987.
- [17] L. Chu, F.L. Qing, *Org. Lett.* 12 (2010) 5060–5063.
- [18] Y. Hu, J. Liu, Z. Lü, X. Luo, H. Zhang, Y. Lan, A. Lei, *J. Am. Chem. Soc.* 132 (2010) 3153–3158.
- [19] Z. Liang, W. Xue, K. Lin, H. Gong, *Org. Lett.* 16 (2014) 5620–5623.
- [20] J. Sun, X. Peng, H. Guo, *Chin. J. Org. Chem.* 35 (2015) 1375–1379.
- [21] E. Baciocchi, M. Crescenzi, *Angew. Chem. Int. Ed.* 29 (1990) 658–659.
- [22] T. Nobuta, A. Fujiya, S. Hirashima, N. Tada, T. Miura, A. Itoh, *Tetrahedron Lett.* 53 (2012) 5306–5308.