RSC Advances

ROYAL SOCIETY OF CHEMISTRY

PAPER

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Cite this: *RSC Adv.*, 2020, **10**, 3946

Efficient synthesis of 1-iodoalkynes *via* Al_2O_3 mediated reaction of terminal alkynes and *N*-iodosuccinimide†

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Received 9th January 2020
Accepted 16th January 2020

DOI: 10.1039/d0ra00251h

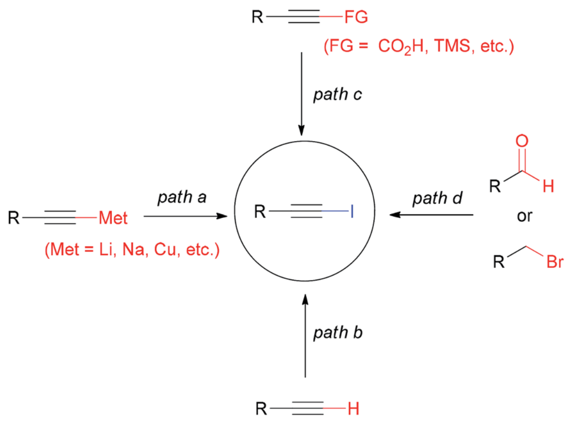
rsc.li/rsc-advances

Iodination of terminal alkynes using *N*-iodosuccinimide (NIS) in the presence of γ-Al_2O_3 was developed to afford 1-iodoalkynes with good to excellent yields (up to 99%). This described approach featured excellent chemoselectivity, good functional group tolerance, and utilization of an inexpensive catalyst.

1-Iodoalkynes are both highly versatile synthons in organic synthesis and valuable building blocks in materials and polymer sciences.[1,2] Traditional procedures for generating 1-iodoalkynes include iodination of metal acetylides (Scheme 1, path a),[3] direct iodination of terminal alkynes (path b),[4] iodination of propiolic acid/trialkylsilylacetylides (path c),[5,6] and a two-step homologation/iodination or elimination sequence from aldehydes or benzylic bromides (path d).[7,8] Among these achievements, path b, the preparation of 1-iodoalkynes from terminal alkynes, is highly desirable. In this regard, various iodinating conditions, including *n*BuLi/I_2,[4a] EtMgBr/I_2,[4b] I_2/DMAP,[4c] NIS/$AgNO_3$,[4d] NIS/BnN_3,[4e] NIS/DBU,[4f] NIS/Ag/C_3N_4,[4g] KI/CuI/PhI$(OAc)_2$/Et_3N,[4h] Me_3SiI/PhI$(OAc)_2$,[4i] CuI/TBAB/Et_3N,[4j] TBAI/Oxone,[4k] KI/TBHP,[4l] TBAI/PhI$(OAc)_2$,[4m] chloramine-B/KI,[4n] KI/Cu_2SO_4/BPDS,[4o] ZnI_2/TBN,[4p] NIS/[Au(AIPr)(NEt_3)][HF_2],[4q] HMBMIBDCI/DBU,[4r] NaI/MeOH/divided cell,[4s] and *N*-iodomorphonine/CuI,[4t] have been reported. However, some of these approaches might suffer from expensive metal catalysts, hazardous or toxic reagents, harsh reaction conditions, or generation of wastes that bring about environmental problems, thereby restricting their practical applications. Though considerable progress has been achieved, the development of efficient and environmentally benign protocols is still required.

It is well recognized that solids can play a vital role in developing new cleaner technologies *via* their capacities to serve as catalysts or support reagents and influence product selectivity, and some books have documented the applications of solids in organic synthesis.[9–11] Aluminum oxide (Al_2O_3) is an inert, odorless and white amorphous material, and has been used as catalyst in various industrial processes for many years.[12,13] But the utilization of aluminum oxide in synthetic organic chemistry especially for halogenation of aromatic compounds and alkynes is very little.[14–18] γ-Aluminum oxide is one of the three common crystal forms of aluminum oxide. Pagni and Kabalka described γ-Al_2O_3 mediated iodination of alkynes and aromatic substrates with I_2 to afford *E*-diiodoalkenes and iodinated aromatic compounds, respectively.[14] Those iodinations did not occur without the activation of γ-Al_2O_3.

N-Iodosuccinimide (NIS) is a well-known iodinating agent and widely used in organic synthesis. Iodination with *N*-iodosuccinimide (NIS) often needs activating reagents. However, any accompanying reagents used along with iodinating reagents should be easily available to exploit more simple and efficient iodination procedure. Inspired by the indispensable role of γ-Al_2O_3 in the iodination of alkynes and aromatic compounds,[14] we envisioned that γ-Al_2O_3 could activate the *N*-iodosuccinimide to generate 1-iodoalkynes from terminal alkynes. Herein, we report the γ-Al_2O_3 mediated direct iodination of terminal



Scheme 1 Approaches towards to 1-iodoalkynes.

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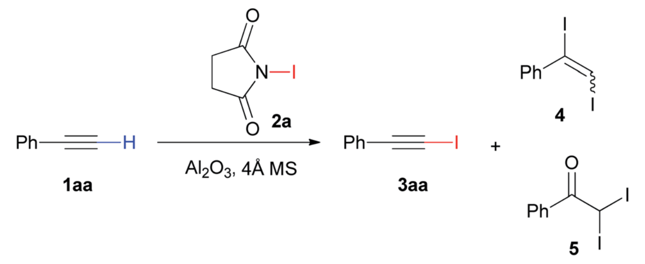
† Electronic supplementary information (ESI) available. See DOI: 10.1039/d0ra00251h

alkynes for the synthesis of 1-iodoalkynes under mild reaction condition.

As shown in Table 1, the initial experiment was carried out by adding *N*-iodosuccinimide (NIS) **2a** (2.2 mmol) to a solution of phenyl acetylene **1aa** (2.0 mmol) and neutral γ-Al_2O_3 (2.6 mmol) in CH_3CN. Surprisingly, the reaction afforded the desired product 1-(iodoethynyl)benzene **3aa** in 90% yield after stirred at 80 °C for 1 h (Table 1, entry 1). To our delight, the reaction could provide 98% yield with the aid of 4 Å MS (entry 2). At the same time, the product **3aa** could be obtained in 95% yield without the help of 4 Å MS when the amount of CH_3CN used in the reaction was reduced from 10 mL to 2 mL (entry 2). Markedly, we obtained a mixture of **1aa**, **3aa**, 1,2-diiodovinylbenzene **4** and 2,2-diiodo-1-phenylethanone **5** in the absent of 4 Å MS and Al_2O_3 (entry 3). In addition, the **3aa** was obtained in 41% yield only in the presence of 4 Å MS (entry 3). Therefore, the Al_2O_3 was indispensable to this iodination reaction (Table S1†). Similarly, high yields of 1-iodoalkyne **3aa** were also obtained when basic and acidic Al_2O_3 were used instead of neutral Al_2O_3 (entry 4–5). Hence, the reaction was further examined in the presence of neutral Al_2O_3. The effort to decrease the reaction temperature to 25 °C only led to low yield (entry 6). Subsequently, we probed different solvents including DMF, THF, EA, MeOH and hexane, but those solvents resulted in poor yields of desired product (entry 7–11). Moreover, the reaction was investigated by varying the amount of Al_2O_3 and NIS to enhance the performance of the reaction (Table 1, entry 12–15). With 1.0 equivalents of NIS, the reaction generated 81% yield of 1-iodoalkyne **3aa** (entry 12). However, when higher or lower amount of Al_2O_3 was used, low yields of the desired product were observed (entry 13–15). Finally, the best conditions to obtain the 1-iodoalkyne **3aa** were treatment of **1aa** with 1.1 equivalents of NIS, 1.3 equivalents of neutral γ-Al_2O_3 and 4 Å MS in CH_3CN at 80 °C for 1 h. With the optimized conditions in hand, we also studied the halogenation of phenyl acetylene **1aa** and used various halogenated reagents, including *N*-bromosuccinimide, 1,3-dibromo-5,5-dimethylhydantoin, pyridinium tribromide and *N*-chlorosuccinimide. These attempts did not provide satisfied results (Scheme S1†).

Table 1 Optimization of the reaction conditions[a]

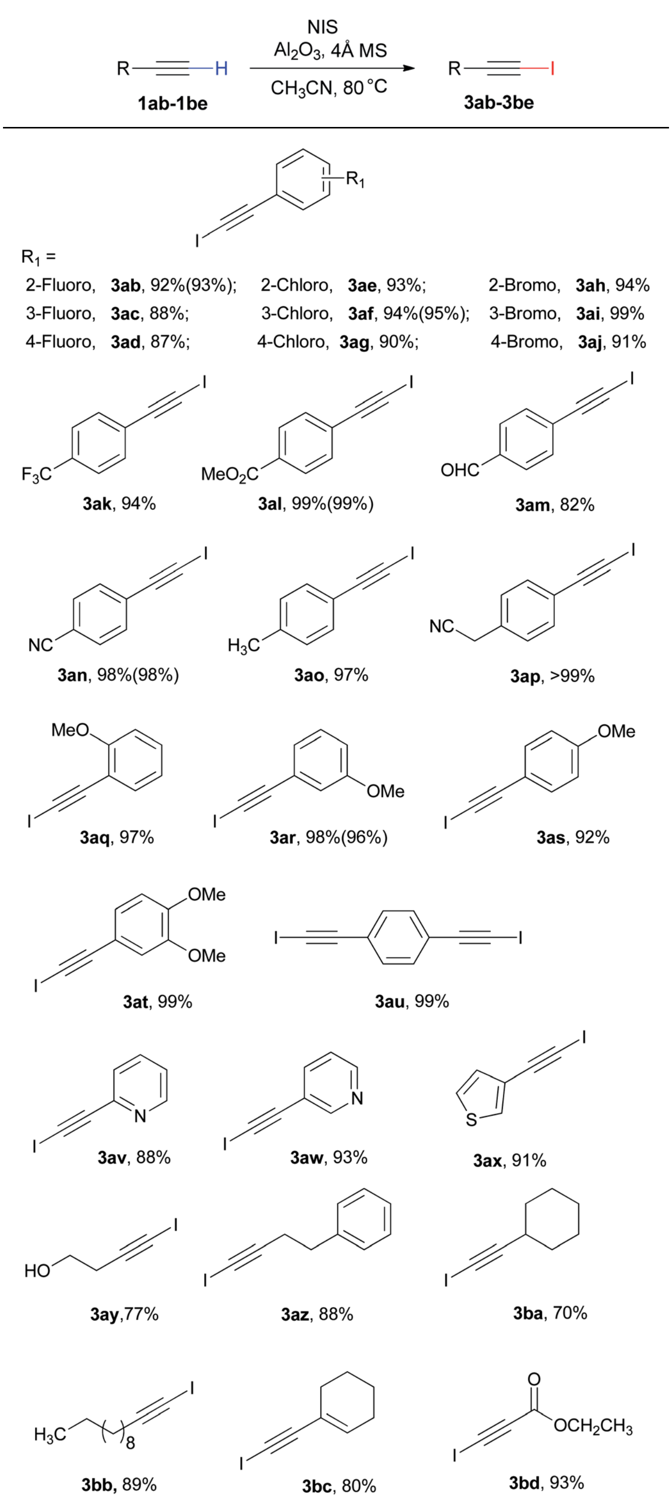


Entry	Solvent	2a (equiv.)	*T* (°C)	Al_2O_3 (equiv.)	Yield[b] (%)
1	CH_3CN	1.1	80	1.3	90[c]
2	CH_3CN	1.1	80	1.3	98(95[h])
3	CH_3CN	1.1	80	—	—[d](41[e])
4	CH_3CN	1.1	80	1.3[f]	96
5	CH_3CN	1.1	80	1.3[g]	97
6	CH_3CN	1.1	25	1.3	42
7	DMF	1.1	80	1.3	63
8	THF	1.1	80	1.3	78
9	EA	1.1	80	1.3	86
10	MeOH	1.1	80	1.3	79
11	Hexane	1.1	80	1.3	62
12	CH_3CN	1.0	80	1.3	81
13	CH_3CN	1.1	80	1.0	84
14	CH_3CN	1.1	80	0.1	70
15	CH_3CN	1.1	80	2.0	91

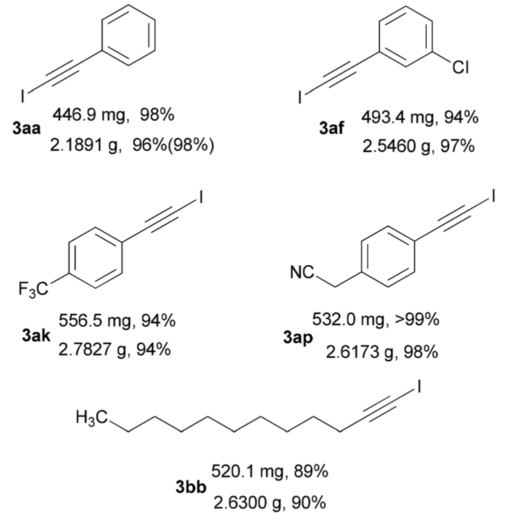
[a] Unless noted otherwise, all reaction were conducted using phenylacetylene **1aa** (2.0 mmol), *N*-iodosuccinimide **2a** (2.2 mmol), 200 mg 4 Å MS, 265.0 mg neutral Al_2O_3 in 10 mL CH_3CN at 80 °C for 1 h. [b] Isolated yield. [c] No 4 Å MS. [d] No Al_2O_3 and 4 Å MS, HPLC analysis of the reaction mixture after reacting at 80 °C for 1 h showed the molar ratio of **1aa** to 1-iodoalkyne **3aa** to 1,2-diiodovinylbenzene **4** to 2,2-diiodo-1-phenylethanone **5** was 8 : 23 24 45. [e] No Al_2O_3. [f] Basic Al_2O_3 was used. [g] Acidic Al_2O_3 was used. [h] The reaction was conducted in 2 mL CH_3CN. EA = ethyl acetate.

Based on the optimized reaction conditions, the generality of the direct iodination of various terminal alkynes was investigated (Table 2). Firstly, a wide variety of aromatic alkynes were firstly evaluated and could react with NIS to afford the corresponding 1-iodoalkynes **3ab–3au** with good to excellent yields. Substrates having both electron-donating (*e.g.* Me, OMe, CH_2CN) and electron-withdrawing (*e.g.* Cl, Br, CF_3, CO_2Me, CN) groups were effectively furnished the desired products. The halogen substituted aromatic alkynes gave the respective 1-iodoalkynes **3ab–3aj** in excellent yields in relation to the position of the halogen on the phenyl ring and the electronegativity of the halogen. Among them, the *meta* bromo substituted alkyne **1ai** gave the best yield (99%). The *para* substituted aromatic alkynes **1ak–1ap** afforded the corresponding products **3ak–3ap** in good to excellent yields. Although the *para* formyl substituted alkyne **1am** yielded the 1-iodoalkyne **3am** in 82% yield, the alkyne **1al** could provide the corresponding product in 99% yield. Notably, the *para* cyanomethyl substituted alkyne **1ap** afforded the desired product in almost quantitative yield (>99%). The dimethoxy substituted alkyne **1at** provided better yield than the single methoxy substituted alkynes (**1aq–1as**). The 1,4-diethynylbenzene **1au** could also be iodinated to generate the desired bisiodinated product in 99% yield. Secondly, the hetero aromatic and aliphatic alkynes were examined under the optimized conditions. The hetero aromatic alkynes delivered the iodination products **3av**, **3aw** and **3ax** in 88%, 93% and 91% yield, respectively. The iodination reaction of aliphatic alkynes underwent smoothly to afford the corresponding products **3ay–3bb** in good yields. The conjugated enyne **1bc** could also react with NIS and gave the anticipated product **3bc** in 80% yield. In addition, the iodination of more reactive alkyne **1bd** was also evaluated and gave the synthetically useful **3bd** in 93% yield. Finally, we also chose five terminal alkynes (**1ab**, **1af**, **1al**, **1an**, **1ar**) to examine the direct iodination requiring a small amount of solvent. The yields of solvent-reduced reaction (values in parentheses) performed in 2 mL CH_3CN were comparable to the yields of the reaction conducted in 10 mL CH_3CN. These results further confirmed that the 4 Å

Table 2 Scope of Al_2O_3/NIS mediated iodination[a]



[a] Reaction conditions: **1** (2.0 mmol), **2a** (2.2 mmol), 4 Å MS (200 mg), Al_2O_3 (2.6 mmol), CH_3CN (10 mL), 80 °C, 1 h. Isolated yields are given. The values in parentheses are the yields of reaction conducted in 2 mL CH_3CN in the absent of 4 Å MS.

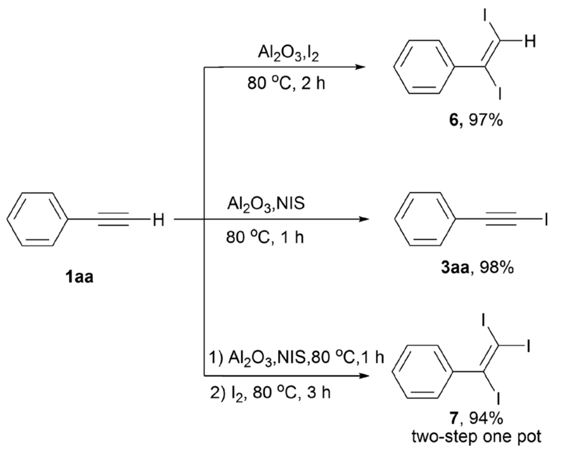


Scheme 2 A Scale-up synthesis of 1-iodoalkynes.

MS was not essential for the Al_2O_3 mediated iodination (Table S1†).

To further explore the potential of this protocol, we conducted the Al_2O_3 mediated direct iodination system for larger-scale synthesis (Scheme 2). The iodination reaction with (10 mmol, 1.0213 g) of phenylacetylene **1aa** in 50 mL CH_3CN afforded 96% yield. Delightfully, when the reaction was performed in 4 mL CH_3CN, the yield could reach to 98% (value in parentheses). Moreover, the iodination of **1af**, **1ak** and **1ap** proceeded smoothly in CH_3CN, producing the corresponding 1-iodoalkynes in 97%, 94% and 98% yield, respectively. Finally, the larger scale reaction of aliphatic alkyne **1bb**, could also generate **3bb** in 90% yield. These excellent results indicated the promise of this direct iodination system for larger-scale preparation of 1-iodoalkynes from terminal alkynes.

As presented in Scheme 3, we also tried to construct mono-, di-, and tri-iodination of terminal alkynes based on direct iodination mediated by γ-Al_2O_3 using phenyl acetylene **1aa** as



Scheme 3 Al_2O_3 mediated chemoselective iodination of phenyl acetylene.

the model substrate. As depicted in the literature,[14b] the di-iodination product **6** could be obtained in 97% yield after stirred at 80 °C for 2 h in the presence of I_2. Combining the NIS and I_2 system in one pot provided the corresponding tri-iodination product **7** in 94% yield.

Finally, we studied the recyclability of Al_2O_3 and 4 Å MS. Al_2O_3 and 4 Å MS could be used as a recyclable catalyst for the direct iodination of phenyl acetylene **1aa** (10 mmol) as it provided 96%, 93% and 88% yield at the first, second and third run, respectively (Fig. S1†). The severe decrease of the yield of the iodination was probably because the active sites of Al_2O_3 were blocked by unknown compounds and the unavoidable loss of solid catalyst during recovery and washing.

Although the detailed mechanism for the γ-Al_2O_3 mediated iodination remains unclear, we proposed that the dehydrated surface of γ-Al_2O_3, which contains partly exposed Al^{3+} and O^{2-}, could serve as an effective medium for electrophilic iodination and greatly increase the chemoselectivity and rate of the reaction. Investigation of the detail mechanism and further applications of this methodology are toward in our laboratory.

Conclusions

In conclusion, we have developed an efficient approach for the synthesis of 1-iodoalkynes *via* γ-Al_2O_3 mediated direct iodination of terminal alkynes in the presence of NIS. Firstly, this protocol has excellent reactivity, high chemoselectivity and good functional group tolerance, and can be used for large-scale preparation of various 1-iodoalkynes. Secondly, with the aid of Al_2O_3 the mono-, di-, and tri-iodination products could be obtained in high yield. Thirdly, the Al_2O_3 and 4 Å MS could be reused for the direct iodination of terminal alkynes at least once without severe decrease of the yield.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

We gratefully acknowledge the Hubei Provincial Department of Education (T201719) for generous financial support.

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