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Electrochemical synthesis of quinazolinone *via* I_2-catalyzed tandem oxidation in aqueous solution†

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The development of protocols for synthesizing quinazolinones using biocompatible catalysts in aqueous medium will help to resolve the difficulties of using green and sustainable chemistry for their synthesis. Herein, using I_2 in coordination with electrochemical synthesis induced a C–H oxidation reaction which is reported when using water as the environmentally friendly solvent to access a broad range of quinazolinones at room temperature. The reaction mechanism strongly showed that I_2 cooperates electrochemically promoted the oxidation of alcohols, then effectively cyclizing amides to various quinazolinones.

The N-heterocycles are key core structures that form the basis of many pharmaceutical, agrochemical and natural products.[1] Among them, quinazolinones are an important motif in several biologically relevant pharmacophores,[2] such as methaqualone which is famous for its effective sedative and hypnotic effects, luotonin A which is a quinazolinone alkaloid with anti-inflammatory effects, and erlotinib which is an anti-tumour agent, and all these compounds contain a quinazoline bond in their backbone (Fig. 1).[3]

Due to their advantageous structures quinazolinones have been widely explored in numerous syntheses.[4] The classical method involves condensation of aldehydes and *o*-aminobenzamides to give aminal intermediates, which then undergo oxidation to yield the final quinazolinone product.[5] Another strategy is to use more benign and readily available alcohols as starting materials.[6] The reaction takes place through a two-step oxidation pathway, where the alcohols are first oxidized to aldehydes, followed by coupling with *o*-aminobenzamides. The catalyst needs to demonstrate high activity and selectivity as the reaction involves dehydrogenation of both the C–H and N–H bonds in one pot. In 2018, Sarma and co-workers[7] demonstrated that a magnetically recoverable iron oxide-carbon dot nanocomposite was an effective catalyst for cyclooxidative tandem synthesis of quinazolinones in aqueous medium using alcohols as starting materials. Furthermore, annulation reactions of *o*-aminoaryl acids may be the most employed strategies, which include the condensation of *o*-aminoaryl acids with amides, nitriles, or acid derivatives plus a nitrogen source.[8] Moreover, the synthesis of quinazolinone involving transition metal catalysed reactions of *o*-haloarylamides with nitriles, or amines[9] and reaction of *o*-halogenated aryl acid with amides[10] have been explored (Scheme 1).

Although the above approaches solved a lot of practical problems, there are still some limitations such as long reaction time, high temperature and by-products. Hence, development of greener, atom economic, synthetic approaches for the preparation of quinazolinones from inexpensive and easily available starting materials under relatively mild conditions is desirable. On one hand, electrochemical-induced direct functionalization has gained significant attention from the synthetic chemistry community due to it being environmentally friendly, and requiring mild conditions, and low-energy irradiation.[11] With electrons as the oxidizing/reducing agent, organic electrosynthesis could offer appropriate alternatives to traditional oxidation or reduction reactions. For example, Zhao and co-workers[12] reported an efficient electrochemical-induced C–H methylthiolation of electron-rich aromatics *via* a three-component cross-coupling strategy. On the other hand, the non-metallic

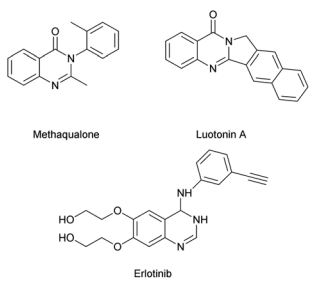

Fig. 1 Bioactive compounds containing quinazolinone skeleton.

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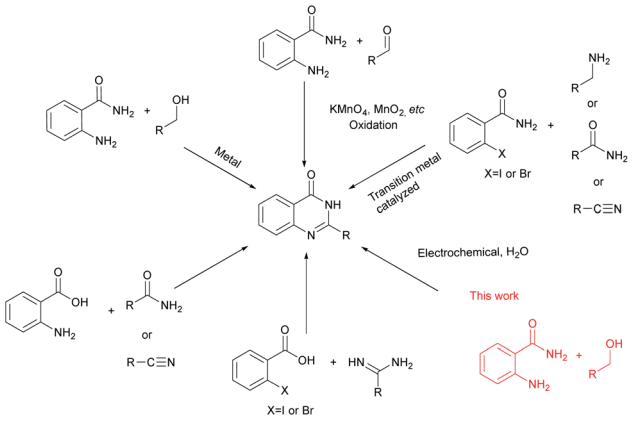


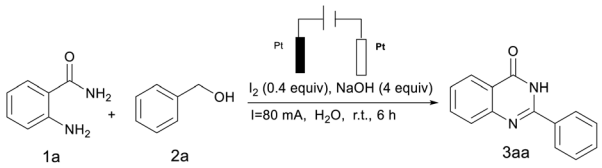
Scheme 1 Methods for the synthesis of quinazolinones.

oxidant, iodine, catalysed C–H oxidation has attracted great interest in recent times due to its low toxicity and because it is inexpensive compared with transition metal catalysts.[13] Therefore, the combination of electrochemical catalysis and non-metallic oxidant iodine is a very feasible means of organic synthesis and does not require use of the historical large doses of iodine. In this research the possibility of combining the two in a one-pot reaction was explored, thus avoiding the isolation of either aldehyde or amine intermediates leading to quinazolinones formed from alcohols as starting materials.

Furthermore, water as a reaction medium is generally considered as an inexpensive, safe, and environmentally benign alternative to organic solvents.[14] Recently, Muthaiah and co-workers[15] demonstrated a catalyst system for the dehydrogenative oxidation of alcohols to carbonyl compounds and dehydrogenative lactonization of diols in water catalyzed by a water-soluble bifunctional iridium complex. In continuation of our work to develop new organic transformations,[16,11f] herein, it is demonstrated that I_2 is an efficient catalyst for a novel, one-pot electrochemical-induced tandem reaction in aqueous solution.

The investigation was initially begun by selecting *o*-aminobenzamide **1a** and benzyl alcohol **2a** as the model substrate to optimize the reaction conditions shown in Table 1. The reaction was initially heated in water using a catalyst: I_2 (0.4 equiv.) and a base: NaOH (4 equiv.) to give the desired product **3aa** with a yield of 92% (Table 1, entry 1). The amount of catalyst used was reduced to one-fifth and the separation rate of the corresponding product was only 42% (Table 1, entry 2). When the reaction mixture was placed in other solvents for 6 h, this reaction takes place with a 73% isolated yield (Table 1, entry 3), and for the sake of environmental protection, water was chosen as a green and energy saving solvent. Poor yields were obtained in the absence of I_2 (Table 1, entry 4). Removal of NaOH from the reaction system led to a disruption reaction that may be due to the unstable or even interrupted current (Table 1, entry 5). Other catalysts such as CuI, TBAI and KI all gave lower reactivity or poor reactivity (Table 1, entries 6–8). Among the bases investigated, NaOH was found to be the best choice (Table 1, entries 9 and 10). Control experiments revealed that no reaction occurred in the absence of current, indicating that it was

Table 1 Optimization of the reaction conditions[a]



Entry	Variations from the standard conditions	Yield[b] (%)
1	**None**	**92**
2	0.1 mmol I_2	42
3	$MeCN/H_2O$ (v/v = 1 : 1) as solvent	73
4	In the absence of I_2	Trace
5	In the absence of NaOH	Trace
6	CuI instead of I_2	19
7	TBAI instead of I_2	26
8	KI instead of I_2	42
9	Cs_2CO_3 instead of NaOH	31
10	KOH instead of NaOH	73
11	No current	Trace
12	Addition of Bu_4NPF_6 as electrolyte	92
13	0.4 mmol I_2 instead of current	28
14	1.0 mmol I_2 instead of current	30
15	C(+)/Pt(−)	64
16	Pt(+)/Cu(−)	16
17	40 mA	71

[a] Standard conditions: undivided cell, Pt anode, Pt cathode, **1a** (0.5 mmol), **2a** (0.6 mmol), I_2 (0.2 mmol), NaOH (2 mmol), H_2O (3 mL), I = 80 mA at room temperature (r.t.) for 6 h. [b] Isolated yield.

essential to the reaction (Table 1, entry 11). Also, no significant improvement in the product yield was observed even when tetrabutylammonium hexafluorophosphate (Bu_4NPF_6) was added as the electrolyte (Table 1, entry 12). An attempt was made to replace the current by increasing the amount of catalyst so that a large number of imines were produced and less than 30% of **3aa** was produced (Table 1, entries 13 and 14). Additionally, other electrode materials such as a graphite anode or Cu cathode have proved less efficient (Table 1, entries 15 and 16). This may be due to the discrepancy in reactivity between the graphite electrode and the Pt electrode which may cause the difference in current density at the anode surface, which results in differing concentrations of electron oxidation products, inhibiting further oxidation of **2a** and decreasing the rate of electron transfer. In addition, the overpotential of hydrogen evolution at the cathode (Cu electrode) is higher than that of Pt, and the aldehydes formed by anodic oxidation may be preferentially reduced to alcohols at the cathode.[17] It was also observed that the reduction in current also leads to a reduction in yield (Table 1, entry 17).

The optimized conditions were then applied to various substrates to extend the scope of the method (Table 2). Various substituted alcohols with different electronic properties and functional groups were tested. It was observed that alcohols bearing electron-donating or electron-withdrawing functional groups could be smoothly converted to the desired products

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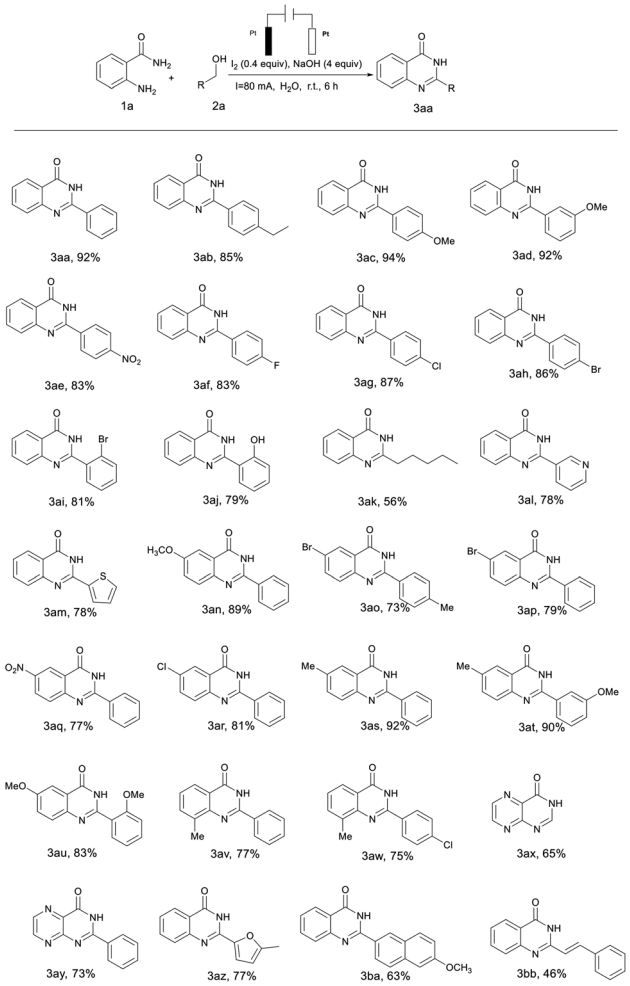
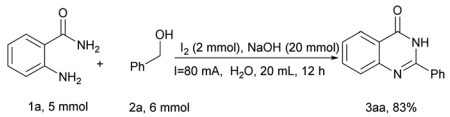
Table 2 Synthesis of quinazolinones from benzyl alcohols and *o*-aminobenzamides[a,b]



[a] Standard Conditions: undivided cell, Pt anode, Pt cathode, **1a** (0.5 mmol), **2a** (0.6 mmol), I_2 (0.2 mmol), NaOH (2 mmol) in H_2O (3 mL), I = 80 mA at room temperature for 6 h. [b] Isolated yield.

with excellent yields of 79–94% (Table 2, **3ab–3aj**). Because of the stability of the electron donor to intermediates and free radical electrons, it was found that the yield of electron-donating substrates (–Et, –OMe) was slightly higher than that of electron-withdrawing substrates (–NO_2, –F, –Cl, –Br, –OH). The highest yield of 94% was obtained with 4-methoxybenzyl alcohol whereas the lowest yield was 79% with 2-hydroxybenzyl alcohol (Table 2, **3ac** and **3aj**). It was obvious that heterocyclic alcohols such as pyridinyl, thienyl and methylfuran were well-tolerated in this catalytic system with 78% and 77% yields (Table 2, **3al**, **3am** and **3az**). Notably, long-chain aliphatic alcohols also yielded a corresponding product, but the yield was dramatically lower than that of aromatic alcohols (Table 2, **3ak**). Furthermore, highly catalytic activities were found in transformations of naphthalenemethanol to the corresponding products (Table 2, **3ba**). When alkenyl alcohol was employed



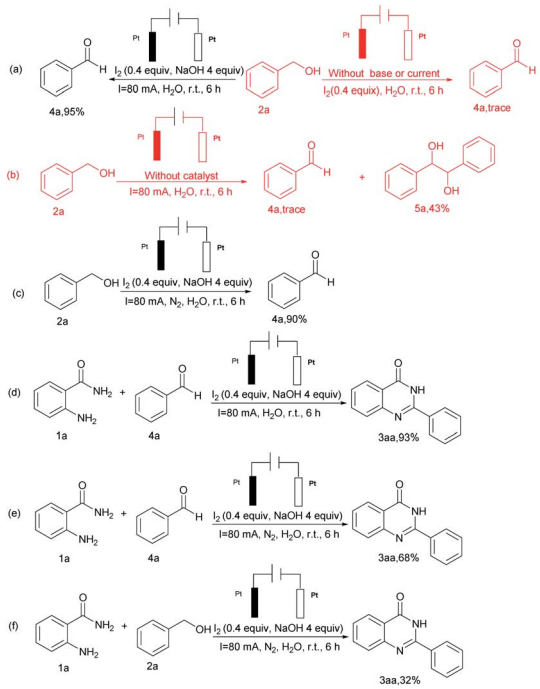
Scheme 2 Gram-scale experiment.

under the same reaction conditions, the product formed only had a 46% yield (Table 2, **3bb**).

Subsequently, the substrates of *o*-aminobenzamide were also expanded (Table 2, **3an–3aw**). Similar to the observation with the alcohol substitution compounds, *o*-aminobenzamides with electron-donating groups (–Me, –OMe) gave relatively higher reactivity than those with electron-withdrawing groups (–NO_2, –F, –Cl, –Br). Whereas when heterocyclic amides were used as the starting substrates, yields of 65% and 73% of the corresponding products were also obtained (Table 2, **3ax** and **3ay**).

In order to demonstrate the practicality of this method, the gram-scale preparation of **1a** (5 mmol) and **2a** (6 mmol) with H_2O (20 mL) under the optimized reaction conditions was performed as shown in Scheme 2, which gave the desired product **3aa** with an 83% yield (1.47 g). This reveals that the new procedure has significant advantages over many current methods for further practical applications.

To exclude the possibility of other reaction pathways, some control experiments were performed (Scheme 3). Firstly, benzyl alcohol **2a** effectively gave benzaldehyde **4a** using aerobic oxidation reactions under standard conditions. In contrast, the absence of either base or current lead to the production of trace amounts of benzaldehyde, furthermore, the lack of catalyst lead to the formation of **5a** (Scheme 3 a and b) with a yield of 43%.



Scheme 3 Control experiments of alcohol oxidation and synthesis of 3aa.

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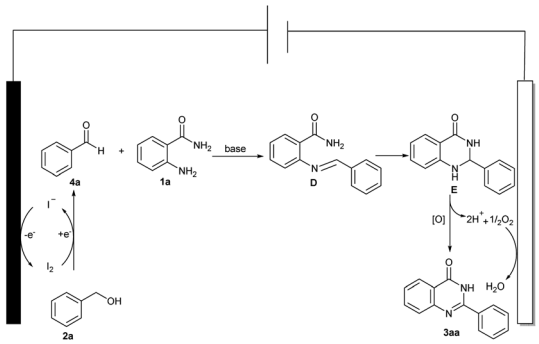
Nevertheless, the yield of **4a** was reduced to 90% when nitrogen was substituted for air (Scheme 3c). Then, the reaction of **1a** and **4a** under the standard conditions effectively gave **3aa** with a high yield of 93% (Scheme 3d), whereas the reaction of *o*-aminobenzamide under a nitrogen atmosphere, resulted in **3aa** with a yield of 68% and 32% (Scheme 3e and f).

To gain an understanding of the reaction mechanism, cyclic voltammetry (CV) was then conducted (Fig. 2). Using H_2O as the solvent, a glass electrode as the working electrode, platinum wire as the opposite electrode, and SCE as the reference electrode with a 0.1 V s^{-1} scanning rate. By comparing curves c, f and g, it was observed that the onset potential of the I_2 oxidation shifted from 1.57 to 1.07 V *vs.* SCE in the presence of substrate **2a**, which was more than that in the presence of NaOH, indicating that I_2 was more likely to react with **b** than disproportionated with NaOH (Fig. 2, curves c, f and g). However, a reduction peak appears in b at 0.43 V, and the reduction peak of I_2 was 0.69 V, indicating that I^- is still carrying out the reduction reaction in the presence of b (Fig. 2, curve c and g). Interestingly, the addition of **1a** and NaOH further decreases the onset potential of I_2 complex oxidation to 0.99 V *vs.* SCE (Fig. 2, curve e). In contrast, the oxidation potential increased when NaOH was added individually, and this may be evidence that NaOH does not interact with **2a** and I_2 (Fig. 2, curve d).



Scheme 4 Proposed mechanism for this transformation.

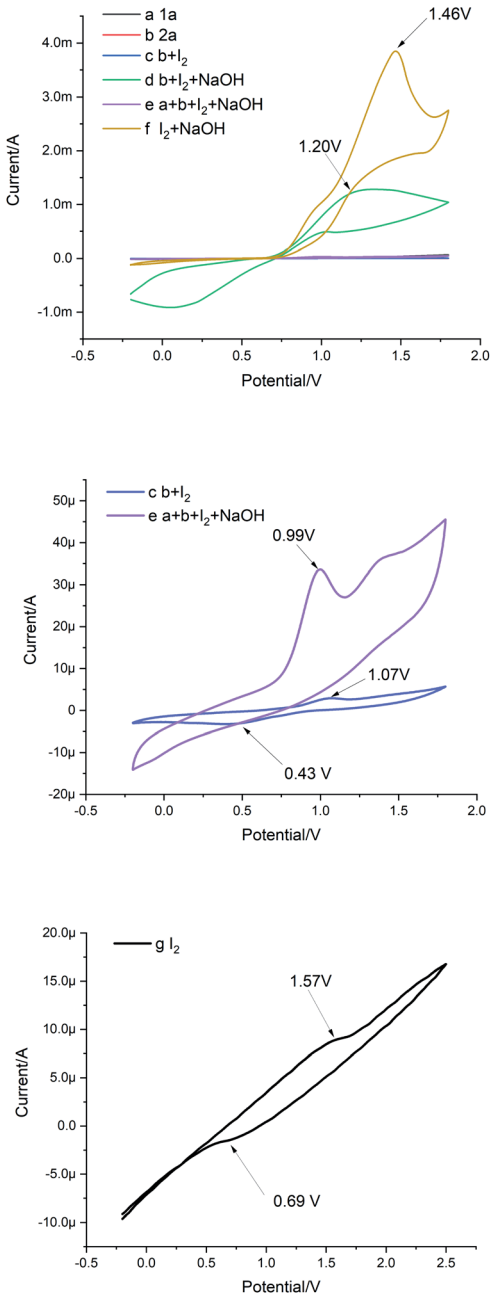
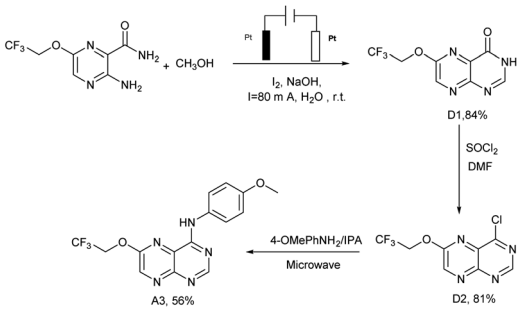


Fig. 2 Cyclic voltammetry measurements were performed at room temperature with standard three electrode systems.

By considering the whole of the experimental findings, a plausible mechanistic pathway for the formation of compound **3aa** is outlined in Scheme 4. First of all, benzyl alcohol **2a** is first oxidized to benzaldehyde **4a** by I_2, which is generated *in situ* on the anode.[6a,18] The iodine undergoes cathodic reduction by regenerating the iodide ion for the catalytic cycle. Next, **4a** can readily react with **1a** to obtain the imine **D**, imine **D** is then converted to **E** in the presence of a base, and thus **E** oxidizes and dehydrogenates to give the desired product **3aa**.[19] Finally, H^+ removed from **E** combines with O_2 to form H_2O at the cathode.[20]

Lots of nuclear nitrogen heterocycles, such as pteridine which is a widely existing aromatic compound, similar to quinazolines, which are tyrosine kinase inhibitors, which have good specificity and inhibitory activity on tumour cells. It is



Scheme 5 Synthesis of *N*-(4-methoxyphenyl)-6-(2,2,2-trifluoroethoxy) pteridin-4-amine (**A3**).

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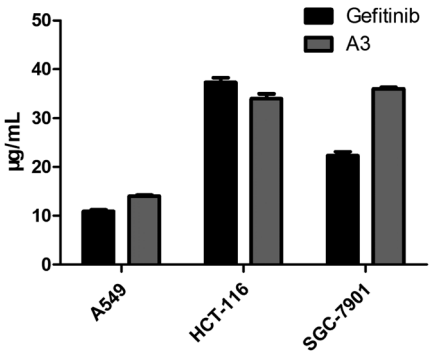

Fig. 3 *In vitro* inhibitory data of target compounds against A549, HCT-116 and SGC-7901 cell line.

thought that many fluorine-containing drugs have been widely used in clinical situations. The introduction of fluorine atoms and fluorine-containing groups into drugs can improve the clinical efficacy of drugs and reduce their side effects. Based on this, the electrocatalytic system was applied to the synthesis of the structure of the trexine and the synthesis of gefitinib analogues. For example, *N*-(4-methoxyphenyl)-6-(2,2,2-trifluoroethoxy)pteridin-4-amine **A3** was synthesized from 3-amino-6-(2,2,2-trifluoroethoxy) pyrazine-2-carboxamide with a yield of 56% (see Scheme 5). The compounds were then tested using the MTT assay and the results are shown in Fig. 3. The **A3** inhibited human non-small-cell lung carcinoma cells, A549, human colon cancer cells, HCT-116, and human gastric cancer cells, SGC-7901 to different degrees. As shown in Fig. 3, the results showed that the IC50 value of **A3** in HCT116 cells (IC50 = 14.79 μg mL^{-1} in A549 cells, IC50 = 34.52 μg mL^{-1} in HCT116 cells, IC50 = 36.44 μg mL^{-1} in SCG-7901 cells) was just surpassed by that of gefitinib.

Conclusions

In summary, it is demonstrated that simple and commercially available I_2 could be used as an electrocatalyst for the selective oxidation of a range of activated C–H bonds in alcohols. It is also demonstrated that I_2 together with electrochemistry is a mild catalyst to efficiently promote C–H oxidation reactions. Oxidative tandem cyclization is of importance in the synthesis of heteropolycyclic skeletons and has a high atom economy. Furthermore, use of both electrocatalysis as a reaction process and water as a green solvent was consistent with the concept of green synthesis. This catalytic protocol avoids using toxic reagents and shows a broad substrate scope, providing consistently good yields of quinazolinones under constant current at room temperature for 6 h. Further extension of this electrochemical protocol to the synthesis of other heterocycles is underway.

Experimental section

General procedure (for Tables 1 and 2)

To a dried 10 mL quartz tube equipped with 80 mA current was added benzamide (0.5 mmol), alcohol (0.6 mmol), I_2 (0.2 mmol), NaOH (2.0 mmol) and water (3 mL). The mixture was stirred at room temperature for 6 h in a 80 mA circuit. After the reaction was completed the solution containing the crude product was concentrated under vacuum, and the residue was purified by column chromatography on a silica gel (petroleum ether/ethyl acetate = 3/1) to give the target product as a white solid.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

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