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Recyclable heterogeneous gold(I)-catalyzed oxidation of internal acylalkynes: Practical access to vicinal tricarbonyls

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ABSTRACT

A highly efficient heterogeneous gold(I)-catalyzed oxidation of internal acylalkynes has been developed using 2,6-dichloropyridine *N*-oxide as the oxidant in dichloromethane (CH_2Cl_2) at room temperature, providing a novel and practical approach for the construction of diverse vicinal tricarbonyls such as α, β-diketoesters, 1,2,3-triketones, and α,β-diketoamides in good to excellent yields. The heterogeneous gold(I) catalyst can be readily obtained *via* a simple preparative procedure from commercially available reagents and recovered by filtration of the reaction mixture and reused up to seven times without significant loss of catalytic efficiency.

Introduction

Vicinal tricarbonyl compounds (VTCs) have drawn considerable attention from synthetic and medicinal chemists in recent years because of their presence in various biologically and pharmaceutically significant compounds, such as the elastase inhibitors YM-47141 and YM-47142 [1], the potent immunosuppressants FK-506 and FR-900525 [2], as well as rapamycin and 29-demethoxyrapamycin [3]. They are also important building blocks in organic synthesis due to their highly electrophilic nature, particularly the central carbonyl, and exhibit significant reactivity toward even rather weak nucleophiles [4]. These compounds have been successfully used for the construction of a variety of carbo- and heterocycles including cyclopentanes [5], pyrroles [6], indoles [7], isoquinolines [8], quinoxalines [9], triazines [10], imidazoles [11], furans [12], and benzofurans [13]. Additionally, applications of VTCs in the total synthesis of natural products have also been reported [14].

Based on the synthetic versatility of VTCs, various methods have been developed for their construction [4]. Conventional synthetic approaches for VTCs include the direct oxidation of β-dicarbonyl compounds [15] or their α-substituted derivatives [16] and oxidative cleavage of the C=C and C = X (X = N, S, P, and I) double bonds of α-functionalized β-dicarbonyl compounds [4a,17]. Among these conventional routes, the oxidation of diazo derivatives of β-dicarbonyl compounds using DMDO [18], *t*-BuOCl [5,19], epoxides [20], or pyridine *N*-oxides [21] appears to be one of the most convenient and frequently used methods. In spite of significant progress made in the development of efficient synthetic methods, these approaches suffer often from disadvantages such as the use of potentially explosive diazo compounds or toxic reagents, a limited substrate scope, harsh reaction conditions, and unsatisfactory yields.

Over the past two decades, catalytic organic reactions involving gold α-oxo carbene intermediates have been widely employed in organic synthesis [22]. Various transformations of the highly electrophilic gold α-oxo carbene intermediates offer an attractive alternative to the reactions of α-diazo carbonyl compounds [23]. Gold α-oxo carbene intermediates can be easily formed through intermolecular oxygen transfer under mild conditions from gold complexes and readily available alkynes with pyridine *N*-oxides as safe oxygen carriers [24]. Recently, the gold(I)-catalyzed oxidation of alkynes and internal acylalkynes with pyridine *N*-oxides as oxidants was reported as a highly efficient method for the synthesis of 1,2-dicarbonyls and 1,2,3-tricarbonyls, respectively, under mild conditions [25]. However, homogeneous gold-catalyzed organic reactions suffer from drawbacks such as the high cost, difficult separation, and non-recyclability of the gold catalysts as well as the decay of cationic gold, which largely limit their applications in large-scale synthesis or in industry. Anchoring homogeneous gold complexes onto a solid support through covalent bond formation is one of the possible ways to address these problems;

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application of the immobilized catalysts in organic transformations could result in easy separation, recovery, and reusability of the gold catalysts, thereby minimizing gold contamination of the target product and waste derived from the reaction workup [26]. The employment of high-surface-area mesoporous materials as catalyst supports is particularly attractive in this regard.

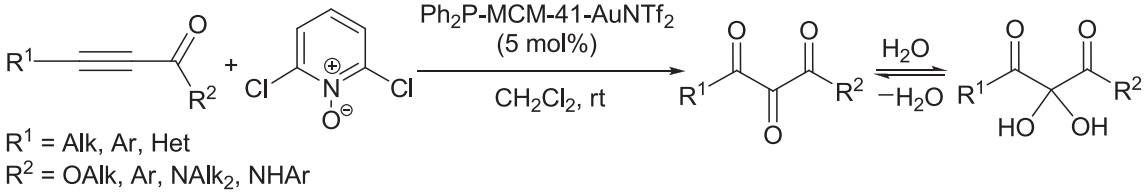
Mesoporous MCM-41 has been widely utilized as an ideal support for the immobilization of homogeneous catalysts owing to its unique properties, such as large pore volume, ultrahigh surface area, homogeneity of the pores, and high thermal stability compared to other solid supports [27]. To date, gold(I) or gold(III) complexes anchored onto MCM-41 have been successfully applied to various organic transformations [28]. Recently, we reported the synthesis of a diphenylphosphine-modified MCM-41-supported gold(I) complex [Ph_2P-MCM-41-$AuNTf_2$] and its successful application to the oxidative ring expansion of 2-alkynyl-1,2-dihydropyridines or -quinolines leading to functionalized azepines or benzazepines [29]. To further expand the applications of this heterogeneous gold(I) catalyst, herein we report a highly efficient heterogeneous gold(I)-catalyzed oxidation of internal acylalkynes leading to vicinal tricarbonyls in good to excellent yields using the Ph_2P-MCM-41-$AuNTf_2$ complex as a recyclable catalyst under mild conditions (Scheme 1).

Results and discussion

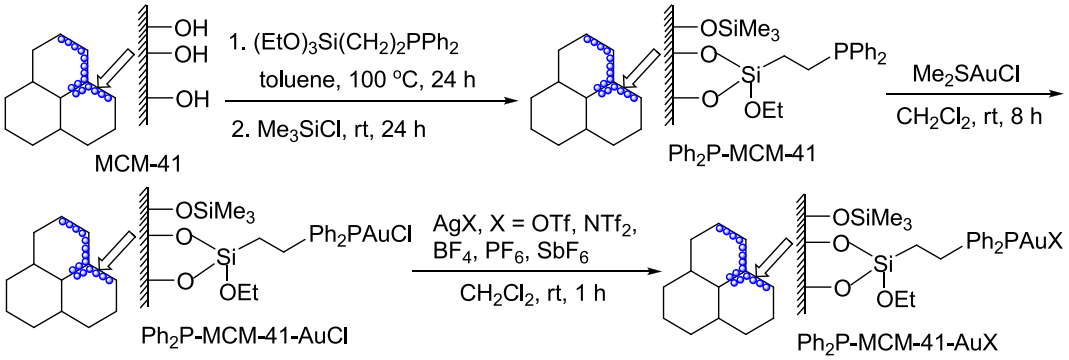
The diphenylphosphine-modified MCM-41-supported gold(I) complexes [Ph_2P-MCM-41-AuX, X = Cl, OTf, NTf_2, BF_4, PF_6, and SbF_6] could be easily prepared from commercially available starting materials *via* a simple procedure as depicted in Scheme 2 [29]. The condensation of mesoporous MCM-41 with 2-(diphenylphosphino)ethyltriethoxysilane in dry toluene at reflux, followed by silylation with Me_3SiCl in dry toluene at room temperature generated the diphenylphosphine-modified MCM-41 material [Ph_2P-MCM-41]. The latter was reacted with Me_2SAuCl in dichloromethane (CH_2Cl_2) at room temperature to give the Ph_2P-MCM-41-AuCl complex. Finally, Ph_2P-MCM-41-AuCl was treated with various silver salts (AgX, X = OTf, NTf_2, BF_4, PF_6, SbF_6) in CH_2Cl_2 at room temperature to afford the diphenylphosphine-modified MCM-41-supported gold(I) complexes [Ph_2P-MCM-41-AuX, X = OTf, NTf_2, BF_4, PF_6, SbF_6] as grey powders.

The diphenylphosphine-modified MCM-41-supported gold(I) complexes [Ph_2P-MCM-41-AuX, X = Cl, OTf, NTf_2, BF_4, PF_6, SbF_6] were then used as the catalysts for the oxidation of internal acylalkynes to vicinal tricarbonyls. Initial experiments with ethyl 3-phenylpropiolate (**1a**) as a model substrate were conducted to optimize the reaction conditions, and the results are listed in Table 1. First, the effect of various pyridine *N*-oxides as oxidants was examined using Ph_2P-MCM-41-$AuNTf_2$ as the catalyst and chlorobenzene as the solvent at 60 °C (Entries 1–5). The bulky and electron-deficient 2,6-dichloropyridine *N*-oxide (**2b**) was found to be the most efficient oxidant, while other pyridine *N*-oxides such as 2-chloropyridine *N*-oxide (**2a**), 4-nitropyridine *N*-oxide (**2c**), 8-methylquinoline *N*-oxide (**2d**), and pyridine *N*-oxide (**2e**) afforded the desired product **3a** in low yields of 20–47%. We next evaluated the influence of different heterogeneous gold(I) catalysts on the model reaction (Entries 6–10). The use of Ph_2P-MCM-41-AuCl as the catalyst did not produce product **3a** and substrate **1a** was recovered in 92% yield (Entry 6). When Ph_2P-MCM-41-AuOTf, Ph_2P-MCM-41-$AuBF_4$, Ph_2P-MCM-41-$AuPF_6$ or Ph_2P-MCM-41-$AuSbF_6$ was used as the catalyst, the reaction afforded product **3a** in 63–77% yield (Entries 7–10), but Ph_2P-MCM-41-$AuNTf_2$ gave the best result (Entry 2). Replacement of chlorobenzene with THF resulted in a significant decrease in the yield of **3a** (Entry 11), whilst the use of DCE, $CHCl_3$, CH_2Cl_2 or $PhCF_3$ as the solvent furnished **3a** in 72–85% yield (Entries 12–15), with CH_2Cl_2 at 40 °C representing the best choice (Entry 14). Gratifyingly, performing the reaction at room temperature in CH_2Cl_2 lead to almost complete conversion of the starting material **1a** within 1 h, providing the desired **3a** in 89% yield (Entry 16). Reducing the amount of the gold catalyst to 2.5 mol% led to a decreased yield of **3a** even when the reaction time was prolonged to 5 h (Entry 17). However, further increasing the amount of the gold catalyst to 10 mol% did not improve the yield of **3a** significantly (Entry 18). When homogeneous $Ph_3PAuNTf_2$ (5 mol%) was used as the catalyst, the desired product **3a** was also isolated in 88% yield (Entry 19), revealing that the catalytic efficiency of Ph_2P-MCM-41-$AuNTf_2$ was comparable to that of $Ph_3PAuNTf_2$. Thus, the best result was achieved with 5 mol% Ph_2P-MCM-41-$AuNTf_2$ and 2,6-dichloropyridine *N*-oxide as the oxidant in CH_2Cl_2 at room temperature for 1 h (Entry 16).

Having established the optimal reaction conditions, the substrate scope of this heterogeneous gold(I)-catalyzed oxidation of internal acylalkynes was investigated. As seen from Table 2, this reaction was quite general for a wide variety of diversely substituted methyl or ethyl 3-phenylpropiolates, and the desired VTCs **3b-w** were generally obtained in good to excellent yields. For example, *para*- or *meta*-substituted methyl or ethyl 3-phenylpropiolates **1b-q** bearing either electron-donating or electron-withdrawing groups gave the corresponding VTCs **3b-q** in 68–90% yield, indicating that the electronic nature of substituents on the benzene ring had limited influence on the reaction. A wide range of substituents such as methyl, *tert*-butyl, methoxy, trifluoromethoxy, fluoro, chloro, bromo, trifluoromethyl, cyano, nitro, ketone and ester functional groups were tolerated. 3,4-Disubstituted 3-phenylpropiolates **1r-s** were also suitable substrates and produced the expected products **3r-s** in 80–95% yield. Sterically hindered *ortho*-substituted 3-phenylpropiolates **1t-w** displayed a relatively lower reactivity than the corresponding *para*-substituted ones and gave the desired VTCs **3t-w** in 55–71% yield. Notably, rigid 4-biphenyl- and bulky 1-naphthyl-substituted propiolates **1x** and **1y** provided the target products **3x** and **3y** in good yields. Heteroaryl-substituted propiolates such as 2- or 3-thienyl-substituted propiolates **1z** and **1a'** also gave the desired VTCs **3z** and **3a'** in 59% and 85% yield, respectively. Next, various alkynyl ketones and amides were examined. Alkynyl ketones **1b'** and **1c'** were successfully converted into the corresponding triketo products **3b'** and **3c'** in 75–82% yield, respectively. Similarly, mono- and di-*N*-substituted alkynyl amides **1d'** and **1e'** afforded the desired VTCs **3d'** and **3e'** in 73–91% yield, respectively. It should

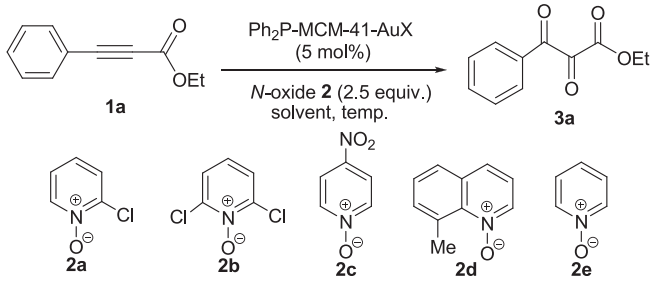


Scheme 1. Heterogeneous gold(I)-catalyzed oxidation of internal acylalkynes towards vicinal tricarbonyls.



Scheme 2. Preparation of the Ph_2P-MCM-41-AuX complexes.

Table 1
Optimization of the reaction conditions.[a]



Entry	X	*N*-oxide	Solvent	Temp. (°C)	Time (h)	Yield **3a** (%)[b]
1	NTf_2	**2a**	PhCl	60	3	47
2	NTf_2	**2b**	PhCl	60	3	82
3	NTf_2	**2c**	PhCl	60	3	28
4	NTf_2	**2d**	PhCl	60	3	23
5	NTf_2	**2e**	PhCl	60	3	20
6	Cl	**2b**	PhCl	60	12	0
7	OTf	**2b**	PhCl	60	3	63
8	BF_4	**2b**	PhCl	60	3	75
9	PF_6	**2b**	PhCl	60	3	66
10	SbF_6	**2b**	PhCl	60	3	77
11	NTf_2	**2b**	THF	60	3	31
12	NTf_2	**2b**	DCE	60	3	77
13	NTf_2	**2b**	$CHCl_3$	60	3	72
14	NTf_2	**2b**	CH_2Cl_2	40	3	85
15	NTf_2	**2b**	$PhCF_3$	60	3	83
16	NTf_2	**2b**	CH_2Cl_2	25	1	89
17[c]	NTf_2	**2b**	CH_2Cl_2	25	5	75
18[d]	NTf_2	**2b**	CH_2Cl_2	25	0.5	90
19[e]	–	**2b**	CH_2Cl_2	25	1	88

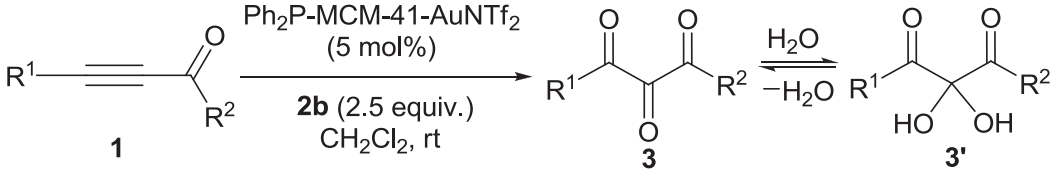
[a] Reagents and conditions: **1a** (0.2 mmol), **2** (0.5 mmol), Ph_2P-MCM-41-AuNTf$_2$ (5 mol%), solvent (0.5 mL). [b] Isolated yield. [c] Ph_2P-MCM-41-AuNTf$_2$ (2.5 mol%) was used. [d] Ph_2P-MCM-41-AuNTf$_2$ (10 mol%) was used. [e] $Ph_3PAuNTf_2$ (5 mol%) was used.

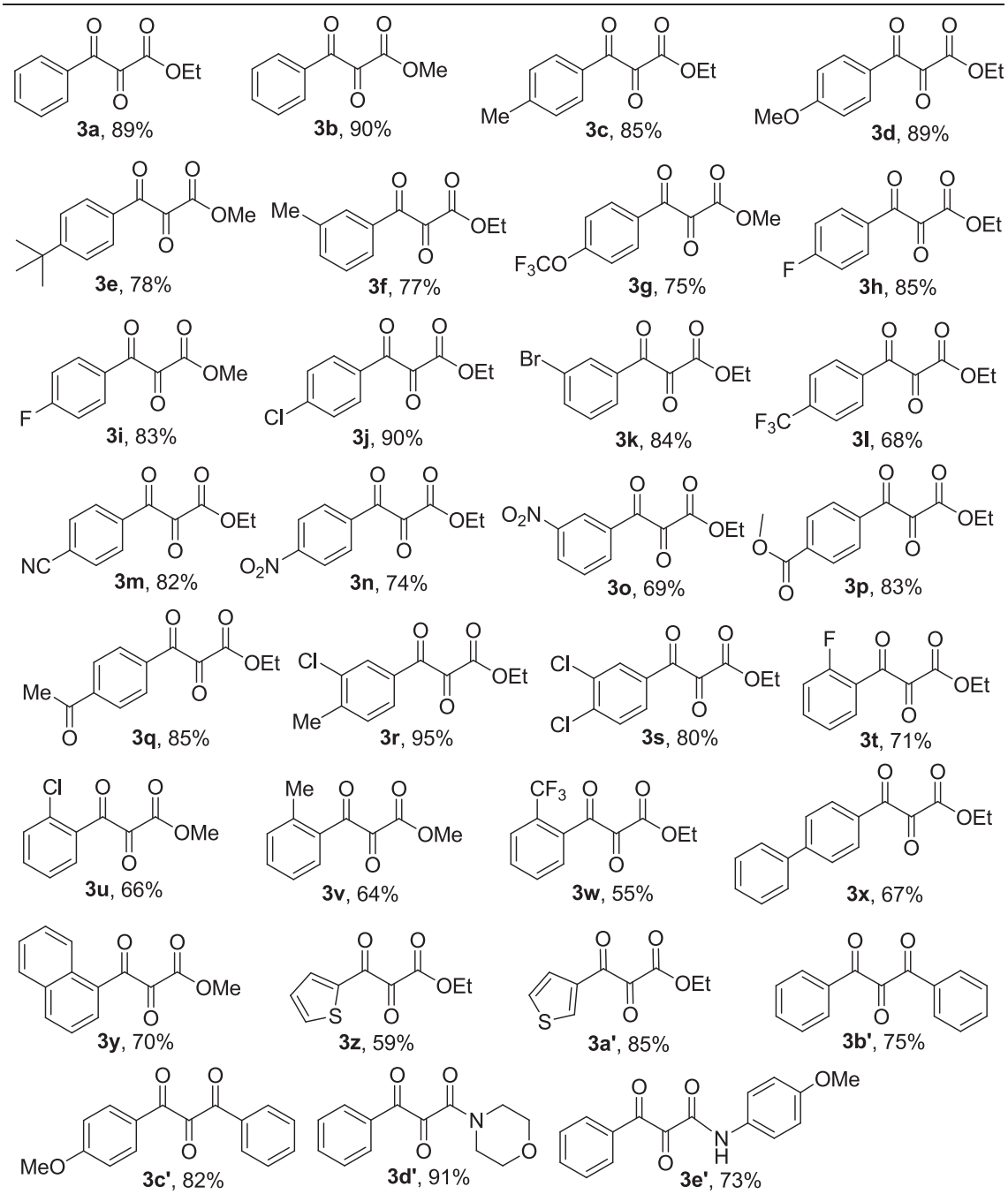
be noted that ester and ketone products were obtained as the hydrate forms **3′** or as mixtures of the keto and hydrate forms with a predominance for the latter. However, in the cases of amide products, keto forms **3** predominate. To further expand the reaction scope, we also examined the reactivity of internal alkynes such as 1,2-diphenylacetylene and 1-phenylhexyne under the standard conditions; unfortunately, the oxidation reaction did not proceed. As reported by Dubovtsev and co-workers, the gold(I)-catalyzed oxidation of internal diarylalkynes to 1,2-diketones requires the use of TfOH (2 equiv.) as an auxiliary reagent to quench the formed 2-methylpyridine and to retain the activity of the gold catalyst [25b].

It should be noted that, like in Zhang's and Deng's works [15a,e], we were also unable to purify aliphatic VTC products by flash chromatography on silica gel, presumably due to facile aldol condensation. To illustrate that alkyl-substituted acylalkynes can also be employed as precursors of VTCs, we performed the one-pot synthesis of quinoxaline derivatives **4** *via* the oxidation of alkyl-substituted acylalkynes **1f'** or **1g'**, followed by condensation with *o*-phenylenediamine (Scheme 3). Treatment of alkyl-substituted acylalkynes **1f'** or **1g'** (0.2 mmol) with **2b** (2.5 equiv.) in $PhCF_3$ in the presence of Ph_2P-MCM-41-AuNTf$_2$ (5 mol%) at room temperature for 1 h, followed by the reaction with *o*-phenylenediamine (1.5 equiv.) at 60 °C for 5 h, provided the desired quinoxaline derivatives **4** in overall yields of 62–75%.

Encouraged by the above results, we next applied this methodology to the one-pot construction of other heterocyclic systems using internal acylalkynes as readily available starting materials. This protocol was proven to be successful for the preparation of valuable acyl-substituted *N*-heterocycles including indoles,

Table 2
Heterogeneous gold(I)-catalyzed synthesis of VTCs.[a,b]

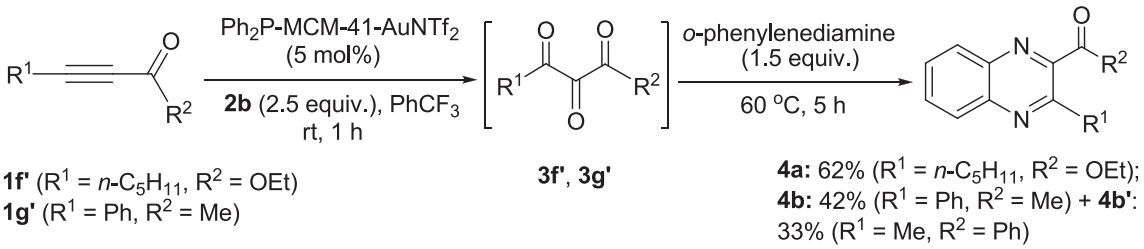




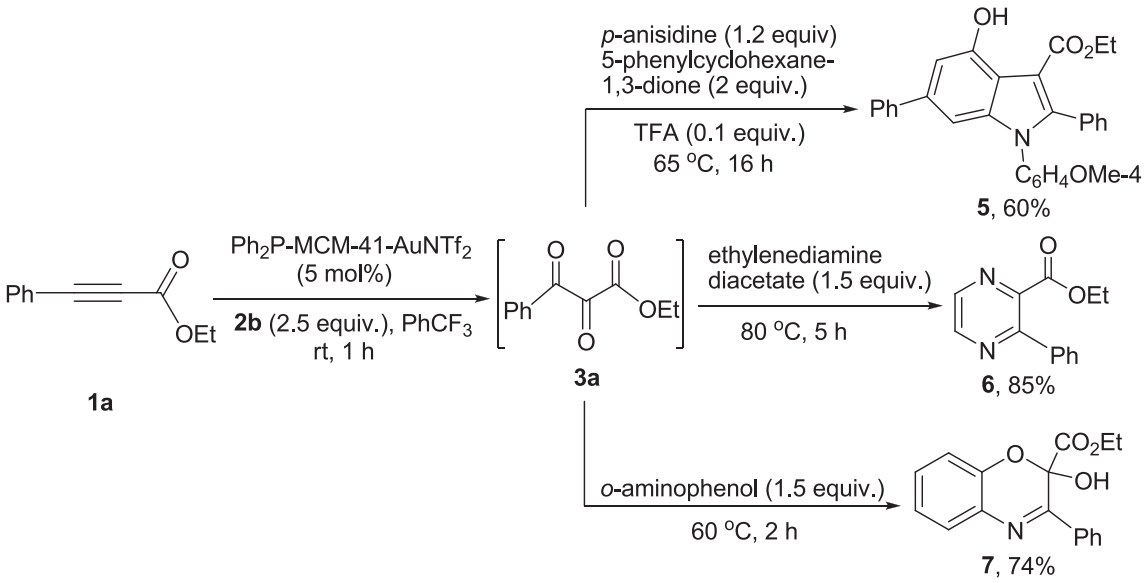
[a] Reagents and conditions: **1** (0.2 mmol), **2b** (0.5 mmol), Ph_2P-MCM-41-$AuNTf_2$ (5 mol%), CH_2Cl_2 (0.5 mL), room temperature, 1 h. [b] Isolated yield.

pyrazines, and benzo[*b*][1,4]oxazines (Scheme 4). These acyl-substituted *N*-heterocycles **5**–**7** were isolated in good overall yields of 60–85%.

To confirm the heterogeneous nature of this oxidation reaction catalyzed by Ph_2P-MCM-41-$AuNTf_2$, the reaction of ethyl 3-phenylpropiolate (**1a**) with **2b** in CH_2Cl_2 was carried out until approximately 40% conversion of **1a** was reached. Then the gold catalyst was removed from the reaction mixture by filtration and the catalyst-free filtrate was stirred at room temperature for an additional 1 h. In this case, no further increase in the conversion



Scheme 3. Heterogeneous gold(I)-catalyzed one-pot synthesis of quinoxalines **4**.



Scheme 4. Heterogeneous gold(I)-catalyzed one-pot synthesis of acyl-substituted *N*-heterocycles 5–7.

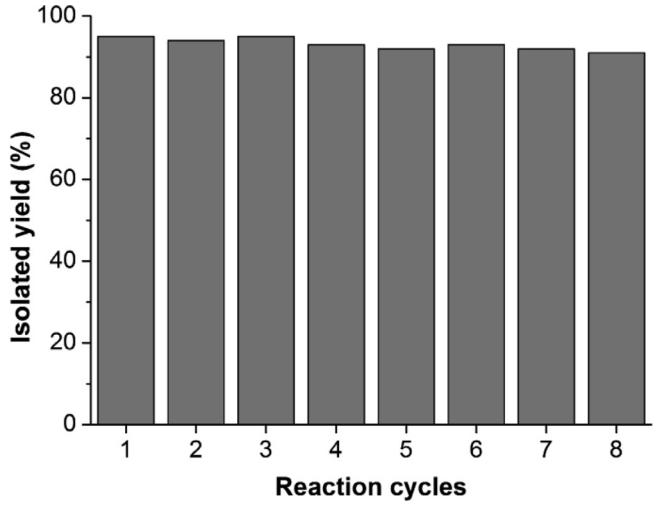


Fig. 1. Recyclability of the Ph_2P-MCM-41-$AuNTf_2$ catalyst.

of **1a** was observed, revealing that leached gold species from the Ph_2P-MCM-41-$AuNTf_2$ catalyst was not related to the observed oxidation. In addition, the filtrate did not contain any gold species (below the detection limit) based on ICP-AES analysis. These results showed that the real catalytic species should be Ph_2P-MCM-41-$AuNTf_2$ and not the leached gold species in the solution, thereby verifying the heterogeneous nature of the oxidation.

From both economic and environmental protection viewpoints, the recovery and recyclability of heterogeneous precious metal catalysts are significant factors for their practical applications. The Ph_2P-MCM-41-$AuNTf_2$ catalyst can be easily separated from the product and recovered through a simple filtration process. We next examined the recyclability of the Ph_2P-MCM-41-$AuNTf_2$ catalyst in the oxidation reaction of ethyl 3-(3-chloro-4-methylphenyl)propiolate (**1r**) with **2b** under the standard conditions (Fig. 1). After the first reaction cycle was completed, the gold(I) catalyst was recovered by filtration of the reaction mixture, followed by washing with acetone and drying at 60 °C *in vacuo* for 1 h. In the recycling experiments, the recovered catalyst was recharged with substrate **1r** for the next reaction run under the same conditions. These results indicated that the Ph_2P-MCM-41-$AuNTf_2$ catalyst still exhibited high catalytic activity even after being reused seven times and the yield of the desired product **3r** was over 91% in eight consecutive runs. In addition, the gold content of the recovered gold(I) catalyst after recycling eight times was found to be 0.37 mmol g^{-1} based on ICP-AES analysis, which revealing negligible gold leaching.

Conclusion

In summary, we have developed a novel, facile and practical method for the synthesis of vicinal tricarbonyls through a recyclable heterogeneous gold(I)-catalyzed oxidation reaction of internal acylalkynes (alkynyl esters, ketones, and amides) with 2,6-dichloropyridine *N*-oxide as the oxidant. The current approach has attractive advantages over the previously reported routes to

VTCs including mild reaction conditions, the use of readily available and safe internal alkynes as substrates, high functional group tolerance, good to excellent yields, and easy recyclability of the expensive gold(I) catalyst. Furthermore, our heterogeneous oxidation methodology can be successfully applied to the one-pot construction of various valuable *N*-heterocycles.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.tetlet.2021.152953.

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