# $Hg(OTf)_2$-Catalyzed Instantaneous Hydration of β- and δ-Hydroxy Internal Alkynes with Complete Regioselectivity

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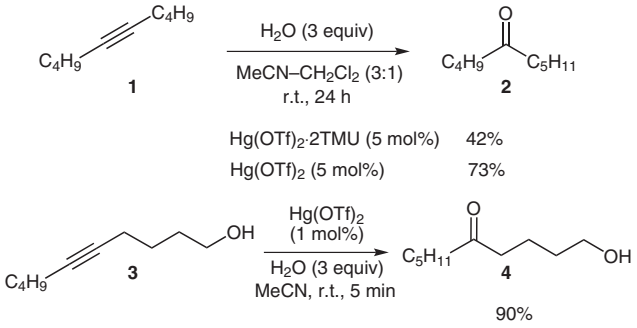
**Abstract:** The hydration of β- and δ-hydroxy internal alkynes catalyzed by $Hg(OTf)_2$ took place instantaneously to give ketones with complete regioselectivity under mild conditions, whereas the hydration of internal alkyne without hydroxy moiety was very slow and gave a mixture of ketones. If the hydroxy group is located more than five carbons from the triple bond it has no significant effect upon the hydration reaction.

**Key words:** $Hg(OTf)_2$, homogeneous catalysis, hydroxy internal alkyne, regioselectivity, neighboring-group effects




**Scheme 1** $Hg(OTf)_2$-Catalyzed hydration of alkyne

Hydration of terminal alkynes giving rise to methyl ketones is an important functional-group transformation in modern organic synthesis.[2] This reaction is usually carried out by heating with a $Hg^{II}$ salt and sulfuric acid.[3] A variety of metal salts such as $Pd^{II}$,[4] $Pt^{II}$,[5] $Au^{III}$,[6] $Au^{I}$,[7] $Rh^{III}$,[8] $Cu^{II}$,[9] and $Fe^{III}$,[10] as well as Brønsted acid[11] have also been investigated. However, these alternative reagents did not yield sufficiently good results to merit adoption as a new standard procedure. A method involving $Ru^{II}$-complex-catalyzed hydration of terminal alkyne leading to aldehyde via anti-Markovnikov addition has also been reported.[12] In 1983 we developed $Hg(OTf)_2$ as a highly efficient olefin cyclization agent.[13] In 2002, the $Hg(OTf)_2$–bistetramethylurea (TMU) complex was found to show highly efficient catalytic activity for the hydration of terminal alkynes leading to methyl ketones under neutral mild conditions.[14] Since then a variety of $Hg(OTf)_2$-catalyzed reactions, such as hydroxylative enyne cyclization, arylyne cyclization, biomimetic tandem cyclization, indole synthesis, furan synthesis, cyclic carbonate synthesis, glycosylation, $S_N2$-reaction, hydroxylative enone synthesis, *E*-selective conjugate ester synthesis, furanoyne cyclization, aryl allyl alcohol cyclization, and *N*-allyl alcohol cyclization have been described.[15a–15n,16] Very recently, the first solid-supported mercuric salt, silaphenylmercuric triflate, was developed that also acts as a powerful catalyst for most $Hg(OTf)_2$-catalyzed reactions.[17]

Here, we investigated $Hg(OTf)_2$-catalyzed hydration of internal alkynes. Very few studies have been conducted on this reaction, mainly due to the poor regioselectivity.[18] We found that $Hg(OTf)_2$-catalyzed hydration of β- and δ-hydroxy internal alkynes takes place almost instantaneously to give γ- and δ-hydroxy ketones, respectively. The reaction proceeds in excellent yield, involving a hydroxy-group participation, with complete regioselectivity. In contrast, hydration of an internal alkyne without hydroxy moiety requires a longer reaction time and affords a mixture of ketones.

When the hydration of dec-5-yne (**1**) was examined using conditions described for the hydration of a terminal alkyne using 3 equivalents of $H_2O$ in the presence of 5 mol% of $Hg(OTf)_2$·2TMU complex in MeCN–$CH_2Cl_2$ (3:1) at room temperature,[14] ketone **2** was obtained in only 42% yield after 24 hours (Scheme 1). Although $Hg(OTf)_2$ itself showed higher reactivity, yield was still 73% after 24 hours. Then we examined the reaction of dec-5-yn-1-ol (**3**) using 1 mol% of $Hg(OTf)_2$ in the presence of $H_2O$ (3 equiv, Scheme 1). The reaction in MeCN–$CH_2Cl_2$ (3:1; the original solvent system employed for the hydration of terminal alkyne)[14] afforded ketone **4** in 79% yield within 10 minutes as a sole product (Table 1, entry 1).[19] Although $MeNO_2$, $CH_2Cl_2$, and THF afforded poor results for this hydration, toluene afforded **4** in 80% yield within 10 minutes. Acetonitrile was the solvent of choice affording **4** in 90% yield after only 5 minutes reaction followed by column chromatography on silica gel (entry 6).[20a] A catalyst loading of 0.1 mol% was also enough to give **4** in 94% yield within an acceptable reaction time. However, a catalyst loading of 0.05 mol% required 20 hours to give **4** in 95% yield.

The reaction involves an equilibrium between *exo*-cyclized product **5** and *endo*-cyclized product **6**, and the favored **5** produces ketone **4** selectively via rate-determining protonation by in situ generated TfOH leading to **7**,

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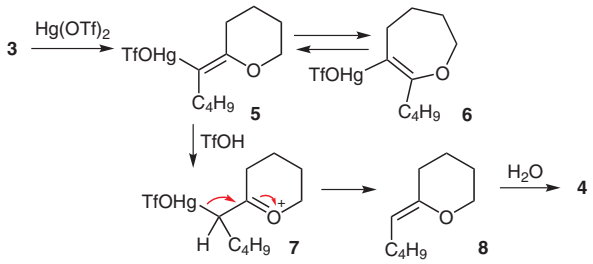
**Table 1** $Hg(OTf)_2$-Catalyzed Hydration of Dec-5-yn-1-ol (**3**)

| Entry | $Hg(OTf)_2$ (mol%) | Solvent | Time | Yield **4** (%)[a] |
|---|---|---|---|---|
| 1 | 1 | MeCN–$CH_2Cl_2$ (3:1) | 10 min | 79 |
| 2 | 1 | $MeNO_2$ | 3 h | 63 |
| 3 | 1 | $CH_2Cl_2$ | 2 h | 76 |
| 4 | 1 | THF | 24 h | 54 |
| 5 | 1 | toluene | 10 min | 80 |
| 6 | 1 | MeCN | 5 min | 90 |
| 7 | 0.1 | MeCN | 2.5 h | 94 |
| 8 | 0.05 | MeCN | 20 h | 95 |

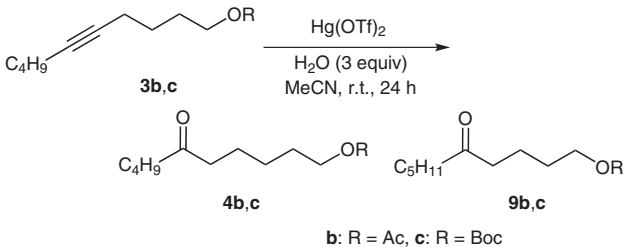
[a] Isolated yield.




**Scheme 2** Mechanism of the selective hydration of **3** to **4**

subsequent demercuration to give **8**, and following hydration.

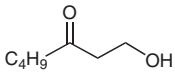
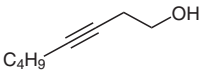
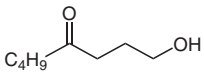
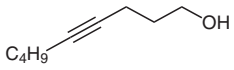
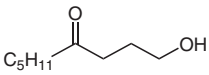
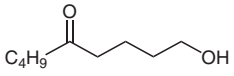
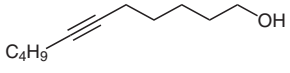
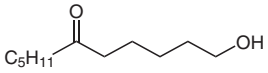
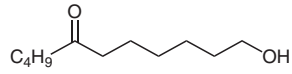
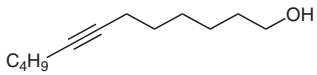
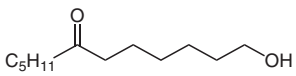
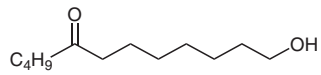
In contrast, the hydration of the protected hydroxyalkyne required long reaction time as is the case of dec-5-yne (**1**). The reaction of acetate **3b** with $H_2O$ (3 equiv) in the presence of 1 mol% of $Hg(OTf)_2$ in MeCN at room temperature for 24 hours afforded a 1:1 mixture of ketones **4b** and **9b** in only trace amounts. The hydration of Boc-protected **3c** also took place very slowly to give a similar result. We reasoned that the observed significant acceleration of the hydration of **3** originated from the participation of the hydroxy moiety thereby producing a cyclic vinyl ether intermediate, such as **5**. By using 5 mol% of $Hg(OTf)_2$, a 1:1 mixtures of **4b** and **9b**, as well as **4c** and **9c** were obtained in 65% and 60% yields, respectively, after 24 hours reaction time (Scheme 3). The ratios were determined by HPLC as well as $^{13}C$ NMR.




**Scheme 3** $Hg(OTf)_2$-Catalyzed hydration of protected 5-alkyn-1-ol

Next we investigated the hydration of a variety of alkynyl-1-ol, and the results are summarized in Table 2. The reaction of hept-2-yn-1-ol (**10**) with 3 equivalents of $H_2O$ in the presence of 5 mol% of $Hg(OTf)_2$ in MeCN at room temperature was very slow, and γ-keto alcohol **11** was ob-

**Table 2** $Hg(OTf)_2$-Catalyzed Hydration of Alkynyl Alcohols[a]

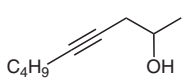
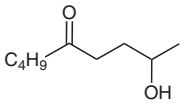
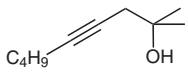
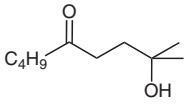
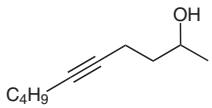
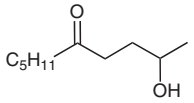
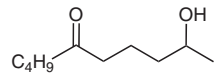
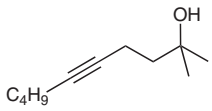
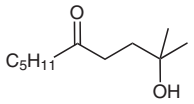
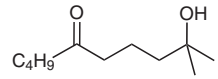
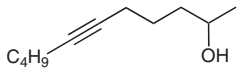
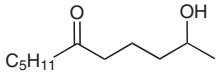
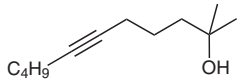
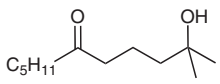
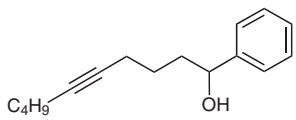
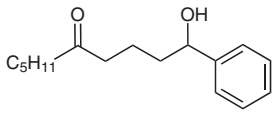
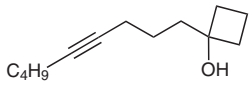
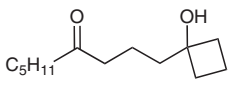
| Substrate | $Hg(OTf)_2$ (mol%) | Time | Product | | Yield (%)[b] |
|---|---|---|---|---|---|
|  **10** | 5 | 24 h |  **11** | | 60 |
|  **12** | 1 | 5 min |  **13**[c] | | 82 |
|  **14** | 1 | 5 min |  **15**[c] |  **16**[c] | 85 (**15**/**16** = 6:1) |
|  **17** | 1 | 24 h |  **18** |  **19** | 75 (**18**/**19** = 3:1) |
|  **20** | 1<br>5 | 24 h<br>24 h |  **21** |  **22** | 51 (**21**/**22** = 1:1)<br>98 (**21**/**22** = 1:1) |

[a] Reaction by using $Hg(OTf)_2$ (1 mol%) and $H_2O$ (3 equiv) in MeCN at r.t.
[b] Yield after column chromatography on $SiO_2$.
[c] Characterized by the derivatization to acetate.

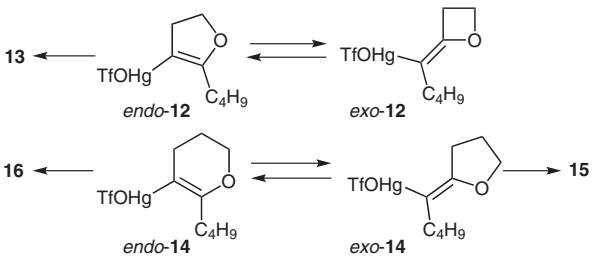
tained in 60% yield after 24 hours.[20b] In sharp contrast, the reaction of oct-3-yn-1-ol (**12**) was completed within 5 minutes to give 1-hydroxyoctan-4-one (**13**) in 82% yield.[20c] Because **13** was obtained as a mixture of keto alcohol and hemiacetal forms, the structure was confirmed by the derivatization to the corresponding acetate upon treatment with acetic anhydride in pyridine. The high regioselectivity should be induced from the favored five-membered ring intermediate *endo*-**12** than the less favored four-membered ring intermediate *exo*-**12** (Scheme 4). The hydration of γ-hydroxyalkyne, non-3-yn-1-ol (**14**), also appeared to take place instantaneously, although in this case a 6:1 mixture of 1-hydroxynonan-4-one (**15**)[20d] and 1-hydroxynonan-5-one (**16**)[20e] was obtained in 85% yield. The result suggested that the equilibrium between *endo*-**14** and *exo*-**14** is energetically comparable to producing both **15** and **16**, respectively. The structures of **15** and **16** were confirmed after derivatization to their acetates. The reaction of dodec-6-yn-1-ol (**17**) was slow, and a 3:1 mixture of **18** and **19** was obtained in 75% yield after 24 hours. Undec-7-yn-1-ol (**20**) also afforded a 1:1 mixture of **21** and **22** in 51% and 98% yields by using 1 mol% and 5 mol% catalyst, respectively, after 24 hours.[21]

The $Hg(OTf)_2$-catalyzed hydration of *sec*- as well as *tert*-β-, γ-, and δ-hydroxyakynes were also examined as shown in Table 3. Hydration of non-4-yn-2-ol (**23**) using 1 mol% of $Hg(OTf)_2$ and 3 equivalents of $H_2O$ in MeCN took place smoothly to give 2-hydroxynonan-5-one (**24**) in

**Table 3** $Hg(OTf)_2$-Catalyzed Hydration of *sec*- and *tert*-Alkynyl Alcohols[a]

| Substrate | Product | | Yield (%)[b] |
|---|---|---|---|
|  **23** |  **24** | | 93 |
|  **25** |  **26** | | 82 |
|  **27** |  **28**[c] |  **29**[c]) | 78 (**28**/**29** = 3:1) |
|  **30** |  **31** |  **32** | 77 (**31**/**32** = 4:1) |
|  **33** |  **34** | | 95 |
|  **35** |  **36** | | 89 |
|  **37** |  **38** | | 87 |
|  **39** |  **40** | | 97 |

[a] Reaction by using $Hg(OTf)_2$ (1 mol%) and $H_2O$ (3 equiv) in MeCN at r.t. for 10 min.
[b] Isolated yield after column chromatography on $SiO_2$.
[c] Characterized after derivatization into acetate.




**Scheme 4** Equilibrium affording **13** selectively and a mixture of **15** and **16**

93% yield selectively within 10 minutes at room temperature via *endo*-mode participation of hydroxy group. Hydration of β-alkynyl *tert*-alcohol **25** using 1 mol% of $Hg(OTf)_2$ also took place smoothly to give **26** in 82% yield selectively within 10 minutes. Hydration of γ-alkynyl *sec*-alcohol **27** and *tert*-alcohol **30** also took place smoothly but produced a mixture of products. Specifically, hydration of **27** and **30** afforded a 3:1 mixture of **28**[20f] and **29**, and a 4:1 mixture of **31** and **32**[20g] in 78% and 77% yield, respectively, based on the energetically comparable *endo*- and *exo*-mode participation. Hydration of δ-hydroxy alkynol **33** afforded δ-hydroxy ketone **34** selectively and instantaneously in 95% yield via *exo*-mode participation.[20h] The *tert*-alcohol, 2-methyldodec-6-yn-2-ol (**35**) was also quickly hydrated in the presence of 1 mol% of $Hg(OTf)_2$ giving rise to **36** in 89% yield selectively. Phenyl-substituted **37** similarly hydrated quickly to give **38** in 87% yield as a sole product. Cyclobutanol derivative **39** also instantaneously provided single keto alcohol **40** in 97% yield.

In conclusion, we found that although the $Hg(OTf)_2$-catalyzed hydration of internal alkynes requires long reaction time, a hydroxy group located in the β-, γ-, and δ-position can significantly accelerate the reaction through the intermediate formation of cyclic enol ethers. Although β-, and δ-hydroxyalkyne selectively generate a single keto alcohol, γ-hydroxyalkyne affords a mixture of keto alcohols via energetically comparable *exo*- and *endo*-mode cyclization. A hydroxy group located in more than five carbons from the alkyne does not participate in the hydration reaction. Indeed, these alkynols behave like an internal alkyne to give a mixture of ketones without regioselectivity. The *sec*- and *tert*-hydroxy groups also accelerate the hydration of alkyne when they are located in the β- and δ-position to give keto alcohols in complete regioselectivity via *endo*- and *exo*-mode participations, respectively.

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## References and Notes

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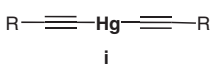
(14) (a) Nishizawa, M.; Skwarczynski, M.; Imagawa, H.; Sugihara, T. *Chem. Lett.* **2002**, 12. (b) When the hydration of terminal alkyne was carried out using $Hg(OTf)_2$ as catalyst, mercuric acetylide (**i**) is formed to some extent, corresponding to catalyst suicide (Figure 1).




**i**

**Figure 1**

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(19) **Typical Experimental Procedure**
To a stirred mixture of dec-5-yn-1-ol (**3**, 280 mg, 2 mmol) and $H_2O$ (108 mg, 6 mmol) in MeCN (6.6 mL) was added a solution of $Hg(OTf)_2$ (0.1 M MeCN soln, 0.2 mL, 0.02 mmol) at r.t., and the mixture was stirred for 5 min at the same temperature. After addition of $Et_3N$ (30 μL) and then brine (6 mL), the organic materials were extracted with $Et_2O$. Dried and concentrated extract was subjected to a column chromatography on $SiO_2$ using hexane and EtOAc (2:1) as an eluent to give 1-hydroxydecan-5-one (**4**, 275 mg, 90% yield) as a colorless oil.[20a]

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(21) All new compounds were fully characterized by spectroscopic methods.

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