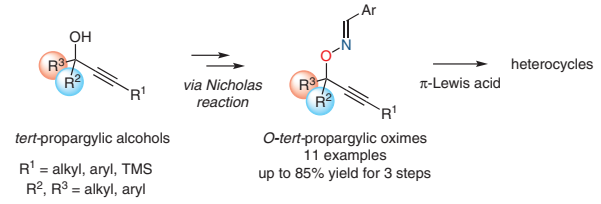
Synthesis I. Nakamura et al. Paper

Efficient Synthesis of *O-tert*-Propargylic Oximes via Nicholas Reaction

Itaru Nakamura[*a] **Keigo Shiga**[b] **Mao Suzuki**[b] **Masahiro Terada**[b]

[a] Research and Analytical Center for Giant Molecules, Graduate School of Science, Tohoku University, 6-3 Aramaki Aza Aoba, Aoba-ku, Sendai 980-8578, Japan
itaru-n@tohoku.ac.jp
[b] Department of Chemistry, Graduate School of Science, Tohoku University, 6-3 Aramaki Aza Aoba, Aoba-ku, Sendai 980-8578, Japan

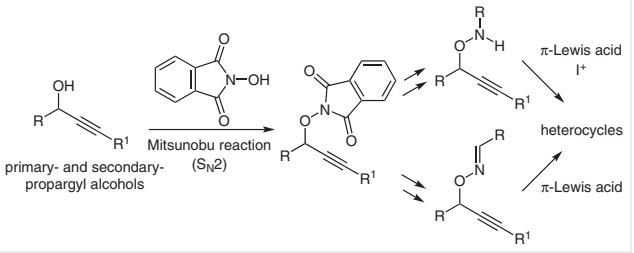


Received: 26.05.2020
Accepted after revision: 09.06.2020
Published online: 21.07.2020
DOI: 10.1055/s-0040-1707191; Art ID: ss-2020-f0290-op

Abstract A synthetic protocol to access *O-tert*-propargylic oximes derived from tertiary propargylic alcohols was established via Nicholas reaction. Thus, $BF_3{\cdot}OEt_2$-mediated reaction between the dicobalt hexacarbonyl complex of *tert*-propargylic alcohols and *p*-nitrobenzaldoxime followed by decomplexation with cerium(IV) ammonium nitrate afforded the corresponding *O-tert*-propargylic oximes in good to high yields. The obtained *O-tert*-propargylic oximes were effectively converted into heterocycles, such as four-membered cyclic nitrones, oxazepines, and isoxazolines, by using π-Lewis acidic catalysts.

Key words Nicholas reaction, alkynes, tertiary alcohols, heterocycles, gold catalysts, spirocycles

N-Propargyloxyamine derivatives have been frequently utilized as a synthetic intermediate of isoxazoline derivatives[1,2] via various transformations, such π-Lewis acidic metal-catalyzed reactions[3] and iodocyclization reactions (Scheme 1).[4] Moreover, we have recently disclosed that *O*-propargylic oximes serve as an intriguing platform for unique heterocycles, such as azete-*N*-oxides[5] and 1,4-oxazepines,[6] by the action of π-Lewis acidic metal catalysts such as Cu, Rh, and Au.[7] In general, *N*-propargyloxyamines derived from primary and secondary propargylic alcohols have been prepared through Mitsunobu reaction between the corresponding propargylic alcohols and *N*-hydroxyphthalimide (NHPI, Scheme 1).[8] However, the protocol is practically inapplicable to propargyloxyamines derived from *tert*-propargylic alcohols **1** due to severe steric repulsion in the S_N2 substitution (Scheme 2a). Thus, it is important to develop efficient and robust approaches to the *O-tert*-propargylic hydroxylamine derivatives for heterocyclic synthesis.[9] Although acid-mediated S_N1-type reactions (Scheme 2b)[10] as well as electrophilic amination reactions (Scheme 2c)[11] have been typically utilized for the synthesis of tertiary alkoxyamine derivatives, our preliminary attempts to synthesize the *O-tert*-propargylic hydroxylamines by these methods from *tert*-propargylic alcohols **1** were unsuccessful, presumably due to instability of the alkyne moiety. Thus, we envisioned that application of the Nicholas reaction[12] would be an effective way to substitute the hydroxy group of the *tert*-propargylic alcohols with the aminooxy group through protection of the alkyne moiety by ligation with two cobalt atoms. Herein, we report an efficient protocol to access *O-tert*-propargylic oximes **2** from the corresponding tertiary-propargyl alcohols **1** via Nicholas reaction (Scheme 2d) and we summarize their reactivity in π-Lewis acidic metal-catalyzed reactions for heterocyclic synthesis.

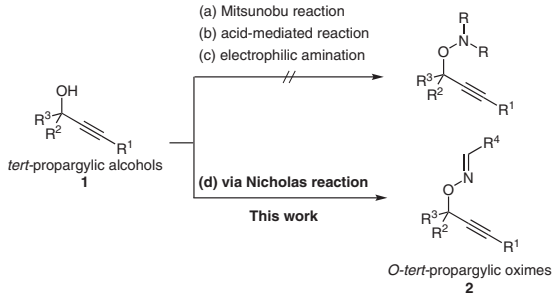


Scheme 1 Preparation of *N*-propargyloxyamines derived from primary and secondary propargylic alcohols

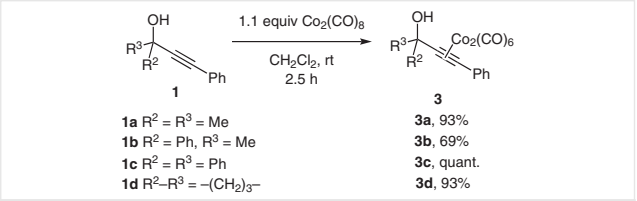
Initially, alkyne **1** were efficiently reacted with $Co_2(CO)_8$ under standard conditions. As represented by the results of **1a–d**, the corresponding dicobalt hexacarbonyl complex **3a–d** were obtained in good to excellent yields (Scheme 3 and Table S1).

Next, Nicholas reactions between the alkyne-dicobalt complex **3** and hydroxylamine derivatives **4** were examined, as summarized in Table 1. The reaction between **3a** and 3 equivalents of the oxime **4a**, derived from *p*-nitro-

Georg Thieme Verlag KG, Rüdigerstraße 14, 70469 Stuttgart, Germany



Scheme 2 Synthesis of *N*-propargyloxyamines derived from tertiary propargylic alcohols via Nicholas reaction

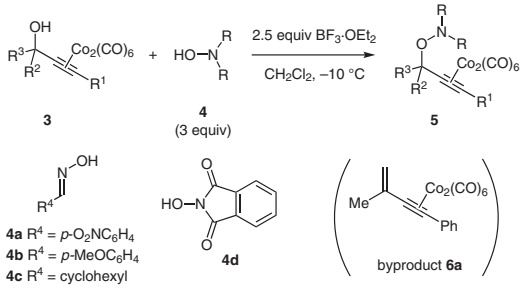


Scheme 3 Ligation of propargylic alcohols **1** with $Co_2(CO)_8$

benzaldehyde in the presence of 2.5 equivalents of $BF_3{\cdot}OEt_2$ at –10 °C, afforded the corresponding *O*-propargylic oxime-dicobalt complex **5aa** in good yield (entry 1). The reaction using either *p*-anisaldoxime (**4b**) or cyclohexanecarbaldoxime (**4c**) resulted in lower chemical yields due to the formation of the elimination byproduct **6a** (entries 2 and 3). The use of NHPI **4d** as a nucleophile was totally inefficient (entry 4). The Nicholas reaction was applicable to various *tert*-propargylic alcohols **3a–j**. For example, substrate **3c**, having geminal diphenyl groups at the propargylic position, was efficiently converted into the corresponding product **5ca** (entry 6), while the use of 5 equivalents of **4a** was effective for the reaction of **3b**, having phenyl and methyl groups (entry 5). Substrates **3d–f**, having a cycloalkyl moiety, reacted with **4a** to afford the desired products **5da–fa**, respectively, in good to acceptable yields (entries 7–9). Substrates **3g** and **3h**, having an aryl group at the alkyne terminus, reacted with **4a** to afford the desired product in excellent yields, irrespective of its electronic character (entries 10 and 11). An alkyl substituent was tolerated at the alkyne terminus (entry 12). In addition, a trimethylsilyl group was tolerated at R^1, affording the desired product **5ja** in good yield (entry 13).

Decomplexation of **5aa**, bearing a *p*-nitrophenyl group at the oxime moiety (R^4), was efficiently promoted by using 4 equivalents of cerium ammonium nitrate (CAN) to quantitatively afford the *O*-*tert*-propargylic oxime **2aa** (Table 2, entry 1; see also the Supporting Information). The propargylic oxime **2ga**, having an electron-rich *p*-anisyl group at the alkyne terminus, was obtained by reducing the loading amount of CAN (2 equiv), due to instability of the substrate **5ga** under the oxidative conditions (entry 2). However, it

Table 1 Nicholas Reaction of **3** with Hydroxylamine Derivatives **4**[a]



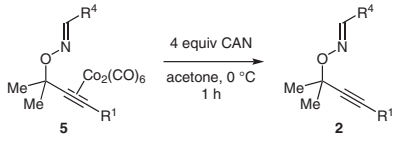
Entry	3	R^1	R^2, R^3	4	Time (h)	5	Yield (%)[b]
1	**3a**	Ph	Me, Me,	**4a**	2.5	**5aa**	91
2	**3a**	Ph	Me, Me,	**4b**	1.5	**5ab**	53
3	**3a**	Ph	Me, Me,	**4c**	0.75	**5ac**	47
4	**3a**	Ph	Me, Me,	**4d**	16.5	**5ad**	<1[c]
5	**3b**	Ph	Ph, Me	**4a**[d]	9	**5ba**	95
6	**3c**	Ph	Ph, Ph	**4a**	1.75	**5ca**	94
7	**3d**	Ph	-$(CH_2)_3$-	**4a**	2	**5da**	75
8	**3e**	Ph	-$(CH_2)_4$-	**4a**	0.5	**5ea**	36
9	**3f**	Ph	-$(CH_2)_5$-	**4a**	2	**5fa**	58
10	**3g**	*p*-$MeOC_6H_4$	Me, Me	**4a**	12.5	**5ga**	>99
11	**3h**	*p*-$F_3CC_6H_4$	Me, Me	**4a**	11.5	**5ha**	>99
12	**3i**	*n*-Hex	Me, Me	**4a**	11.5	**5ia**	94
13	**3j**	Me_3Si	Me, Me	**4a**	1	**5ja**	64

[a] The reaction of **3** (0.4 mmol) and **4** (0.8 mmol) was carried out in the presence of $BF_3{\cdot}OEt_2$ in CH_2Cl_2 at –10 °C for 0.5–16.5 h.
[b] Isolated yield.
[c] Compound **6a** was obtained in 70% yield.
[d] Compound **4a** (2.0 mmol, 5 equiv) was used.

should be noted that *O*-*tert*-propargylic oximes with a variety of aryl groups at the alkyne terminus are potentially accessible from **2ja** via desilylation followed by Sonogashira reaction (entry 5). Substrate **5ia**, having an alkyl group at the alkyne terminus, was readily decomplexed to afford the desired product **2ia** in excellent yield (entry 4), whereas **5ac**, bearing an alkyl group at the oxime moiety, was not converted into the desired product, but suffered decomposition under the reaction conditions (entry 6).

Obtained *O*-*tert*-propargylic oximes **2** were employed for π-Lewis acidic metal-catalyzed reactions to synthesize heterocycles. For example, the reaction of the propargylic oximes **2ca** in the presence of a catalytic amount of $[CuCl(cod)]_2$ at 80 °C afforded the corresponding four-membered cyclic nitrone (azete-*N*-oxide) **7ca** in good yield (Table 3, entry 1). The reaction proceeds via [2,3]-rearrangement from propargylic oxime **2** to *N*-allenylnitrone **9** via the vinylmetal intermediate **8** followed by 4π-electrocyclization. The substrate **2ba**, having phenyl and methyl groups at the propargylic position, afforded a ca. 1:1 mixture of *E*/*Z* stereoisomers at the *exo*-olefin moiety (entry 2).

Table 2 Decomplexation of **5**[a]



Entry	5	R^1	R^4	2	Yield (%)[b]
1	**5aa**	Ph	*p*-$O_2NC_6H_4$	**2aa**	quant
2[c]	**5ga**	*p*-$MeOC_6H_4$	*p*-$O_2NC_6H_4$	**2ga**	25
3	**5ha**	*p*-$F_3CC_6H_4$	*p*-$O_2NC_6H_4$	**2ha**	58
4	**5ia**	*n*-Hex	*p*-$O_2NC_6H_4$	**2ia**	91
5	**5ja**	Me_3Si	*p*-$O_2NC_6H_4$	**2ja**	66
6	**5ac**	Ph	Cy	–	<1

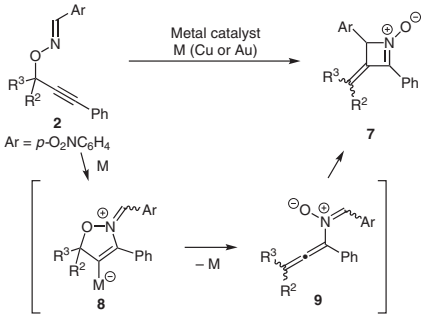
[a] The reaction of **5** (0.1 mmol) was carried out in the presence of CAN (0.4 mmol) in acetone at 0 °C for 1 hour.
[b] Isolated yield.
[c] CAN (0.2 mmol) was used.

The chemical yield of **7aa**, derived from **2aa** having two methyl groups at the propargylic position, was improved by using $(PPh_3)AuNTf_2$ instead of $[CuCl(cod)]_2$ (entries 4 vs. 3). We previously reported that Au catalysts did not promote [2,3]-rearrangement of *O*-propargylic oximes derived from secondary alcohols, presumably because the ring-opening process from **8** to **9** involves cleavage of the C–Au bond, which is much stronger than the C–Cu bond due to the relativistic nature of the gold atom.[5] Accordingly, the present results indicate that substitution by two alkyl groups at the propargylic position facilitates the ring-opening process even in the gold-catalyzed reaction.

We have previously reported that the Cu-catalyzed reaction between *O*-secondary propargylic oxime and electron-deficient olefins, such as *N*-methylmaleimide **10**, proceeded through [2,3]-rearrangement, [3+2] cycloaddition between the *N*-allenylnitrone **9** that was generated in situ and the maleimide, and [1,3]-oxygen rearrangement from the nitrogen atom to the allene center carbon, affording the corresponding 1,4-oxazepines (Scheme 4). Thus, the copper-catalyzed cascade reaction was applied to the *O*-*tert*-propargylic oxime **2aa**. The reaction with *N*-methylmaleimide **10** proceeded at 80 °C in dioxane, affording the corresponding oxazepine **11aa** in good yield with excellent *anti*-selectivity.[6]

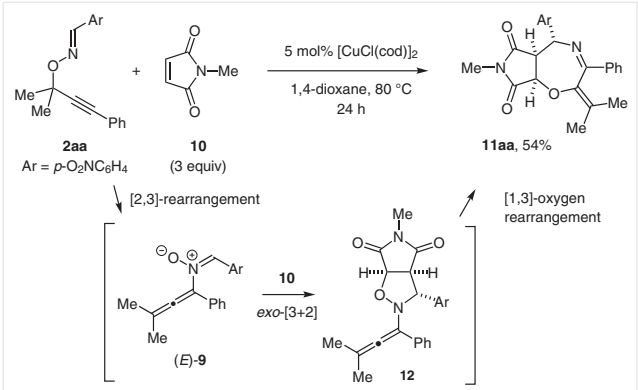
Moreover, the gold-catalyzed reaction of **2aa** in methanol gave isoxazoline **12** in good yield (Scheme 5). In particular, the reaction of **2da**, bearing a cyclobutyl moiety at the propargylic position, was efficiently converted into the corresponding spirocyclic isooxazoline **12da**.[13] The reaction proceeds via alcoholysis of the cyclized vinylgold intermediate **8′** with rapid protodeauration in methanol. In fact, the reaction involved formation of the acetal **13**, supporting the proposed mechanism.

Table 3 Synthesis of Azete-*N*-oxides **7** by Cu- and Au-Catalyzed Reactions of **2**[a]

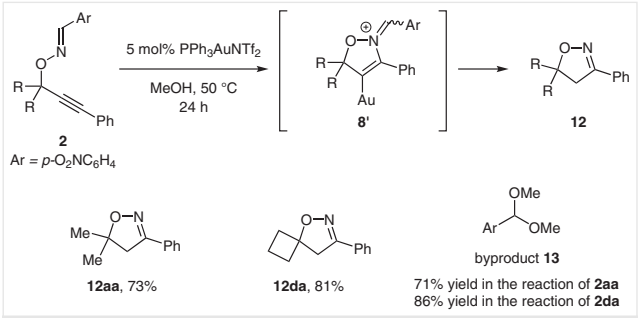


Entry	2	R^2, R^3	Catalyst (mol%)	7	Yield (%)[b]
1	**2ca**	Ph, Ph	$[CuCl(cod)]_2$ (5)	**7ca**	85
2	**2ba**	Ph, Me	$[CuCl(cod)]_2$ (5)	**7ba**	86[c]
3	**2aa**	Me, Me	$[CuCl(cod)]_2$ (5)	**7aa**	30
4	**2aa**	Me, Me	$(PPh_3)AuNTf_2$ (5)	**7aa**	56
5[d]	**2ea**	-$(CH_2)_4$-	$(PPh_3)AuNTf_2$ (5)	**7ea**	52

[a] Reaction conditions for Cu catalysis: $[CuCl(cod)]_2$ (0.01 mmol) in CH_3CN (0.2 mL) at 80 °C for 44 h. For Au catalysis: $(PPh_3)AuNTf_2$ (0.01 mmol) in 1,2-dichloroethane (0.2 mL) at 70 °C for 9 h.
[b] Isolated yield.
[c] A 44:56 mixture of *E*/*Z* isomers was obtained.
[d] At 80 °C.



Scheme 4 Cu-catalyzed reaction between *O*-*tert*-propargylic oxime **2aa** and *N*-methylmaleimide **10**



Scheme 5 Au-catalyzed cyclization of **2** to 2-isoxazolines **12**

In conclusion, we have developed an efficient approach to *O-tert*-propargylic oximes derived from tertiary propargylic alcohols by using the Nicholas reaction. Given that the oximes serve as a platform for catalytic rearrangement reactions and also as an equivalent of propargyloxyamines in π-acidic metal catalysis, the present protocol is useful for the synthesis of highly functionalized heterocycles.

^{1}H and ^{13}C NMR spectra were recorded on a JEOL JNM-ECS400 (400 MHz for ^{1}H and 100 MHz for ^{13}C) spectrometer. Chemical shifts are reported in ppm relative to $CHCl_3$ (for ^{1}H, δ 7.26), and $CDCl_3$ (for ^{13}C, δ 77.00). Infrared (IR) spectra were recorded on a JASCO FT/IR- 4100 spectrophotometer. High-resolution mass spectra analysis was performed on a Bruker Daltonics APEX III FT-ICR-MS spectrometer and Bruker Daltonics solariX FT-ICR-MS spectrometer at Research and Analytical Center for Giant Molecules, Graduate School of Science, Tohoku University. Flash column chromatography was performed on silica gel 60N (Merck 40-63 µm or Kanto 40-50 µm). Analytical thin layer chromatography (TLC) was performed on Merck pre-coated TLC plates (silica gel 60 F254).

Acetone, hexane, CH_3CN, 1,4-dioxane, 1,2-dichloroethane, and methanol were purchased form Fujifilm Wako Pure Chemical Corporation. CH_2Cl_2 was purchased from Kanto Chemical Co., Inc. $Co_2(CO)_8$, $BF_3{\cdot}OEt_2$, and CAN were purchased from TCI. These reagents were used as received. $[Cu(cod)]_2$ and $(PPh_3)AuNTf_2$ were prepared according to reported procedures.[14,15] $CDCl_3$ was purchased from Merck. All air- and moisture-sensitive manipulations were performed under argon atmosphere using oven-dried glassware, including glovebox techniques.

Compound 3a

To a suspension of $Co_2(CO)_8$ (5.6 g, 16.5 mmol) in CH_2Cl_2 (150 mL) in a 300 mL three-neck flask was added **1a** (15 mmol) at room temperature. The mixture was stirred at room temperature for 3 h, then the solvents were removed in vacuo, the crude product was purified by silica gel column chromatography using hexane/CH_2Cl_2 (1:2) as eluent to afford **3a**.

Yield: 6.18 g (13.9 mmol, 93%).

IR (neat): 3606, 3535, 3082, 2988, 2933, 2475, 2089, 2041, 2001, 1606, 1573, 1482, 1459, 1443, 1377, 1362, 1312, 1231, 1153, 1106, 1073, 1030, 998, 954, 918, 828, 761, 736, 691, 672, 621 cm^{-1}.

^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.61 (m, 2 H), 7.35 (m, 3 H), 1.72 (s, 6 H).

^{13}C NMR ($CDCl_3$, 100 MHz): δ = 199.59, 164.68, 137.71, 129.73, 128.83, 127.76, 106.99, 96.10, 91.69, 73.40, 32.44.

HRMS (FD): *m*/*z* [M + Na]$^+$ calcd for $C_{17}H_{12}Co_2O_7$: 468.9139; found: 468.9139.

Compound 5aa

To a mixture of **3a** (6.18 g, 13.9 mmol) and oxime **4a** (6.94 g, 41.8 mmol) in CH_2Cl_2 (140 mL) was added $BF_3{\cdot}OEt_2$ (4.42 mL, 35.2 mmol) dropwise at –10 °C. After stirring at –10 °C for 2.5 h, the reaction was quenched with aqueous $NaHCO_3$ solution and the mixture was extracted with CH_2Cl_2. The organic layer was washed with water and brine and dried over anhydrous sodium sulfate. After solvents were removed in vacuo, the residue was purified by silica gel column chromatography using hexane/EtOAc (5:1) as eluent to obtain **5aa**.

Yield: 7.57 g (12.7 mmol, 91%).

IR (neat): 3428, 3413, 3370, 3359, 2989, 2937, 2376, 2362, 2347, 2334, 2323, 2090, 2051, 2021, 1601, 1587, 1523, 1482, 1442, 1377, 1345, 1140, 1109, 962, 852, 795, 762, 691 cm^{-1}.

^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.19–8.15 (m, 2 H), 8.11–8.10 (m, 1 H), 7.58–7.54 (m, 4 H), 7.36–7.34 (m, 3 H), 1.88 (s, 1 H).

^{13}C NMR ($CDCl_3$, 100 MHz): δ = 199.58, 148.01, 138.64, 138.25, 129.73, 128.75, 127.50, 123.85, 102.45, 92.61, 83.65, 46.24, 29.15.

HRMS (FD): *m*/*z* [M + Na]$^+$ calcd for $C_{24}H_{16}Co_2N_2O_9$: 616.9412; found: 616.9412.

Compound 2aa

To **5aa** (4.87 g, 8.16 mmol) in acetone (81.6 mL) in a 200 mL round-bottom flask was added CAN (17.9 g, 32.6 mmol) at 0 °C. After stirring at 0 °C for 5 h, the reaction was quenched with water and the mixture was extracted with ether. The organic layer was washed with water and brine and dried over Na_2SO_4. After removing solvents in vacuo, the residue was purified by silica gel column chromatography using hexane/EtOAc (10:1) as eluent to obtain **2aa** in analytically pure form.

Yield: 2.52 g (8.16 mmol, quant).

IR (neat): 2997, 2949, 1598, 1589, 1519, 1491, 1443, 1421, 1409, 1341, 1305, 1245, 1215, 1174, 1154, 1102, 1070, 1028, 1013, 947, 851, 795, 757, 690, 642, 622 cm^{-1}.

^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.25–8.23 (d, *J* = 8.7 Hz, 2 H), 8.17 (s, 1 H), 7.82–7.80 (d, *J* = 8.7 Hz, 2 H), 7.45–7.43 (m, 2 H), 7.31–7.29 (m, 3 H), 1.74 (s, 6 H).

^{13}C NMR ($CDCl_3$, 100 MHz): δ = 148.29, 147.56, 138.40, 131.78, 128.35, 128.20, 127.81, 123.92, 122.62, 90.45, 84.87, 83.79, 77.89, 35.06, 14.04.

HRMS (FD): *m*/*z* [M + Na]$^+$ calcd for $C_{18}H_{16}N_2O_3$: 331.1053; found: 331.1053.

Funding Information

This work was supported by JSPS KAKENHI Grant Number JP16H00996 in Precisely Designed Catalysts with Customized Scaffolding and JP20H02731 (Grant-in-Aid for Scientific Research (B)) from MEXT Japan.

Supporting Information

Supporting information for this article is available online at https://doi.org/10.1055/s-0040-1707191.

References

(1) Taylor, R. D.; MacCoss, M.; Lawson, A. D. G. *J. Med. Chem.* **2014**, *57*, 5845.
(2) (a) Lipshutz, B. H. *Chem. Rev.* **1986**, *86*, 795. (b) Baraldi, P. G.; Barco, A.; Benetti, S.; Pollini, G. P.; Simori, D. *Synthesis* **1987**, 857.
(3) (a) Knight, D. W.; Proctor, A. P.; Clough, J. M. *Synlett* **2010**, 628. (b) Yeom, H.-S.; Lee, E.-S.; Shin, S. *Synlett* **2007**, 2292. (c) Nakamura, I.; Onuma, T.; Kanazawa, R.; Nishigai, Y.; Terada, M. *Org. Lett.* **2014**, *16*, 4198.

(4) (a) Foot, O. F.; Knight, D. W.; Low, A. C. L.; Li, Y. F. *Tetrahedron Lett.* **2007**, *48*, 647. (b) Okitsu, T.; Sato, K.; Potewar, T. M.; Wada, A. *J. Org. Chem.* **2011**, *76*, 3438.
(5) (a) Nakamura, I.; Araki, T.; Zhang, D.; Kudo, Y.; Kwon, E.; Terada, M. *Org. Lett.* **2011**, *13*, 3616. (b) Nakamura, I.; Kudo, Y.; Araki, T.; Zhang, D.; Kwon, E.; Terada, M. *Synthesis* **2012**, *44*, 1542.
(6) Nakamura, I.; Kudo, Y.; Terada, M. *Angew. Chem. Int. Ed.* **2013**, *52*, 7536.
(7) (a) Nakamura, I.; Zhang, D.; Terada, M. *J. Am. Chem. Soc.* **2010**, *132*, 7884. (b) Nakamura, I.; Okamoto, M.; Sato, Y.; Terada, M. *Angew. Chem. Int. Ed.* **2012**, *51*, 10816. (c) Nakamura, I.; Sato, Y.; Takeda, K.; Terada, M. *Chem. Eur. J.* **2014**, *20*, 10214. (d) Nakamura, I.; Gima, S.; Kudo, Y.; Terada, M. *Angew. Chem. Int. Ed.* **2015**, *54*, 7154. (e) Gima, S.; Nakamura, I.; Terada, M. *Eur. J. Org. Chem.* **2017**, 4375. (f) Gima, S.; Shiga, K.; Terada, M.; Nakamura, I. *Synlett* **2019**, *30*, 393. (g) Shiga, K.; Terada, M.; Nakamura, I. *Chem. Sci.* **2019**, *10*, 5283.
(8) (a) Somanadhan, B.; Loke, W.-K.; Sim, M.-K.; Go, M.-L. *Bioorg. Med. Chem.* **2002**, *10*, 207. (b) Proctor, A. J.; Beautement, K.; Clough, J. M.; Knight, D. W.; Li, Y. *Tetrahedron Lett.* **2006**, *47*, 5151.
(9) (a) Gaudemer, F.; Gaudemer, A. *Tetrahedron Lett.* **1980**, *21*, 1445. (b) Sabbasani, V.; Lee, D. *Org. Lett.* **2015**, *17*, 4878.
(10) (a) Reddy, C. R.; Radhika, L.; Kumar, T. P.; Chandrasekhar, S. *Eur. J. Org. Chem.* **2011**, 5967. (b) Ren, Z.; Mo, F.; Dong, G. *J. Am. Chem. Soc.* **2012**, *134*, 16991. (c) Kang, T.; Kim, H.; Kim, J. G.; Chang, S. *Chem. Commun.* **2014**, *50*, 12073.
(11) (a) Choong, I. C.; Ellman, J. A. *J. Org. Chem.* **1999**, *64*, 6528. (b) Sun, R.; Li, Y.; Lü, M.; Xiong, L.; Wang, Q. *Bioorg. Med. Chem. Lett.* **2010**, *20*, 4693.
(12) (a) Lockwood, R. F.; Nicholas, K. M. *Tetrahedron Lett.* **1977**, *18*, 4163. (b) Teobald, B. J. *Tetrahedron* **2002**, *58*, 4133. (c) Diaz, D. D.; Betancort, J. M.; Martin, V. S. *Synlett* **2007**, 343.
(13) Kotha, S.; Deb, A. C.; Lahiri, K.; Manivannan, E. *Synthesis* **2009**, 165.
(14) Chi, K. M.; Shin, H.-K.; Hampden-Smith, M. J.; Duesler, E. N.; Kodas, T. T. *Polyhedron* **1991**, *10*, 2293.
(15) Mézailles, N.; Ricard, L.; Gagosz, F. *Org. Lett.* **2005**, *7*, 4133.