Ion-Functionalized Silver(I) Carboxylates: Synthesis and Application in Ru-Catalyzed Olefin Metathesis Reaction

Xiao-Lu Chen^{*a*,†}, Xiang-Yu Li^{*a*,†}, Shuai-Shuai Li^{*a*}, Xue Bai^{*a*}, Tao Li^{*a*}, Erukala Yadaiah Goud^{*b*}, Chong-Min Zhong^{*a*,*}, and Ya-Jie Zuo^{*c*,**}

^a College of Chemistry and Pharmacy, Northwest A&F University, Yangling, 712100 China
 ^b College of Life Science, Northwest A&F University, Yangling, 712100 China
 ^c College of Natural Resources and Environment, Northwest A&F University, Yangling, 712100 China
 e-mail: *zhongcm@nwsuaf.edu.cn; **641935795@qq.com

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Abstract—The Grubbs–Hoveyda catalysts have a wide range of applications in catalyzed formation of the carboncarbon double bonds. In this study, several imidazolium- functionalized or pyridinium-functionalized silver carboxylates have been prepared, and their reaction with Grubbs–Hoveyda 2^{nd} generation (G–H 2^{nd}) catalyst leads a new type of processes. Activity and selectivity of the catalysts have been preliminarily evaluated using cross olefin metathesis reactions.

Keywords: silver(I) carboxylates, ionic-function, ruthenium catalyst, olefin metathesis

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INTRODUCTION

The Ru-catalyzed olefin metathesis reaction leading to formation of carbon-carbon bonds is widely employed in chemical synthesis [1–4]. Design of well-defined ruthenium alkylidenes via modifying both steric and electronic properties of the ligands leads to development of a wide range of versatile olefin metathesis catalysts. Recently, replacement of chlorine ions in Grubbs's ruthenium catalysts by other anionic ligands has received close attention. Grubbs and co-workers reported the first example of replacing chlorine in Ru(=CH-CH=CPh₂). $Cl_2(PPh_3)_2$ with trifluoroacetate [5]. Shortly after, the first stable trifluoroacetate coordinated Grubbs 1st generation catalyst [6] was prepared. However, the catalyst was only moderately active for internal alkene, probably due to its dimer structure. Many other electron-withdrawing ligands, such as trifluoromethanesulfonate, fluoroalkyl carboxylates and pentafluorophenolate [7-10], and trialkoxysilyl substituted carboxylate [11] were used in reactions with Grubbs-Hoveyda 2nd generation catalyst. However, these catalysts usually were less stable/ active than G–H 2nd catalyst and gave E/Z mixtures in cross metathesis reactions. The effects of sulfonate and phosphate ligands on E/Z selectivity [12] has been

studied. Highly Z-selective catalysts were developed by Grubbs [13], Hoveyda [14], and Wang et al. [15]. Besides, the *E*/Z-selectivity can be achieved by changing the structure of the anionic ligands [16]. However, most of the reported catalysts with replaced chlorine showed low or moderate activity. Therefore, design of more active and stereoselective Ru catalysts for olefin metathesis is still quite desirable.

Previously we studied magnetic nano-particles supported G–H 2nd type catalysts [17, 18] and found that ionic functionalized Hoveyda carbene ligand could greatly improve activity of the supported catalysts. However, examples of ionic functionalized chalcogen anionic ligand coordinated Ru catalysts were quite rare [19, 20]. In this study, we report the synthesis of imidazoliumfunctionalized and pyridinium-functionalized and partially fluorinated Ag(I) carboxylates and their preliminary application in G–H 2nd catalyzed cross metathesis reactions.

RESULTS AND DISCUSSION

The commercially available amido-containing ionic liquids are usually impure and cannot be used in preparation of silver(I) carboxylates. The ionic liquids **3** and **6** were synthesized according to the reported procedure [21] as presented in Scheme 1.

[†]Deceased.

Scheme 1. Synthesis of amido-functionalized ionic liquids 3 and 6.



3a: M = K, X = PF₆⁻,
$$t = 2$$
 h, 100%
3b: M = Ag, X = BF₄⁻, $t = 10$ min, 100%

Synthesis of ionic-functionalized silver carboxylates. Synthetic approach to silver carboxylates is outlined in Scheme 2. Generally, ionic liquids **3a** and **3b** were reacted with hexafluoroglutaric anhydride (7). The resulting raw carboxylic acids were used directly in the following reaction with silver oxide. Addition of Ag₂O to a solution of **8a** or **8b** in acetonitrile upon the appropriate procedures gave the corresponding solid products **9a** and **9b**. Silver carboxylate **11** was prepared in a similar way.

Commercially available hydroxyl-functionalized imidazolium ionic liquid 12 reacted with hexafluoroglutaric anhydride 7 in THF to give ion-functionalized carboxylic acid 13 (Scheme 2, part c). Addition of Ag_2O to the solution of 13 in acetonitrile gave silver carboxylate 14. ¹H and ¹³C NMR spectra of compound 14 indicated the presence of carboxylic acid 13 (14 : 13 = 75.2 : 24.8), which might be produced from light induced decomposition of 14 in the course of running NMR.

Tuble 1. Synthesis duta for compounds 150 150	Table	1.	Synthesis	data for	compounds	15a–15c
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Molar ratio	Products
G-H 2nd : $9a = 1 : 1$	G–H 2nd : 15a = 1 : 7.89
G-H 2nd : 9b = 1 : 1	G–H 2nd : 15b = 1 : 5.90
G–H 2nd : 11 = 1 : 1	G–H 2nd : 15c = 1 : 8.55

Synthesis and activity of G–H II type catalysts. Activity of G–H 2nd in the processes with ionfunctionalized carboxylates 9, 11 and 14 was studied (Scheme 3, Table 1). According to ¹H NMR spectra compounds 9b and 11 contained some fluorine-containing impurities, and compound 15 contained a small quantity of unreacted G–H 2nd.

 Table 2. Cross olefin metathesis reaction catalyzed by 15a–

 15c and G–H 2nd



Scheme 2. Synthesis of ionic-functionalized silver(I) carboxylates.





Cross olefin metathesis reaction. The catalytic activity of compounds **15a–15c** (containing G–H 2nd) was tested using a cross olefin metathesis reaction of allylbenzene with methyl acrylate (Table 2). For comparison, G–H 2nd catalyst was also used to catalyze the reaction under the same reaction conditions. According to the accumulated data (Table 2), activity of **15a–15c** bearing ion-functionalized carboxylate anion was somewhat lower than that of G–H 2nd catalyst. The G–H 2nd and **15a–15c** catalysts demonstrated no Z/E selectivity (Z : E = 1.0 : 1.0).

EXPERIMENTAL

The chemicals were purchased from the commercial chemical companies and used without additional

purification. The solvents were dried according to the standard methods. All reactions were carried out under the atmosphere of argon. IR spectra were recorded on a FTIR spectrophotometer. ¹H, ¹³C, and ¹⁹F NMR spectra were measured on a Bruker Avance spectrometer (500 MHz) using MeOD, CD₃CN or DMSO as solvents and TMS as a standard. Melting points were measured by RY-IG melting point instrument. Elemental analysis was carried out on a Vario EL III instrument.

Synthesis of compounds 1 and 4. 1,2-Dimethylimidazole (150 mmol) was loaded to a 100 mL doublenecked round-bottom flask which was evacuated and refilled with Ar three times. Anhydrous acetonitrile (50 mL) and 2-bromethylamine hydrobromide (25 mmol)

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Scheme 3. Synthesis of compounds 15a-15c.



were added and the mixture was refluxed. In 6 h another batch of 2-bromethylamine hydrobromide (25 mmol) was added to the flask (totally 100 mmol). Upon completion of the reaction, the solid was separated by centrifugation, washed with small quantity of ethanol 5 times and dried under vacuum to give the pure product 1.

3-(2-Aminoethyl)-1,2-dimethyl-1*H***-imidazol-3-ium bromide hydrobromide (1)** [21]. White solid, yield 40%, mp 220–225°C. IR spectrum, v, cm⁻¹: 3466, 3397 (NH₂). NMR spectrum, δ , ppm: 7.63 d (1H, J = 1.9 Hz, =CH), 7.56 d (1H, J = 1.9 Hz, =CH), 4.54 t (2H, J = 6.5 Hz, CH₂), 3.85 s (3H, CH₃), 3.49 t (2H, J = 6.5 Hz, CH₂), 2.72 s (3H, CH₃). ¹³C NMR spectrum, δ_{C} , ppm: 147.10, 124.45, 122.26, 46.30, 39.78, 35.90, 10.57. Found, %: C 28.27; H 5.62; N 13.69. C₇H₁₅Br₂N₃. Calculated, %: C 27.93; H 5.02; N 13.96.

Compound **4** was synthesized according to the above method used for **1. 1-(2-Aminoethyl)pyridin-1-ium bromide hydrobromide (4).** White solid, yield 40%, mp 202–208°C. IR spectrum, v, cm⁻¹: 3464, 3420 (NH₂). ¹H NMR spectrum, δ , ppm: 9.17 d (2H, J = 5.6 Hz, H_{Py}), 8.71 t (1H, J = 7.8 Hz, H_{py}), 8.23 t (2H, J = 7.1 Hz, H_{py}), 5.08 t (2H, J = 6.4 Hz, CH₂–C), 3.75 t (2H, J = 6.4 Hz, CH₂–C). ¹³C NMR spectrum, $\delta_{\rm C}$, ppm: 148.13, 146.67, 130.02, 59.20, 40.59. Found, %: C 29.35; H 4.80; N 9.87. C₇H₁₂Br₂N₂. Calculated, %: C 29.61; H 4.26; N 9.86.

Synthesis of compounds 2 and 5. The suspension of compound 1 (10 mmol) in methanol (15 mL) was stirred for 5 min, and then NaOH (10 mmol) was added. After stirring the mixture for 2 h, anhydrous dichloromethane (15 mL) was added and the mixture was stirred for 2 h more. The precipitate (NaCl) was filtered off and washed 3 times with acetonitrile. The combined filtrates were

concentrated, and white solid of NaCl was precipitated again. The solid was filtered and washed 3 times with acetonitrile. The above procedure was carried on as long as NaCl was precipitating.

3-(2-Aminoethyl)-1,2-dimethyl-1H-imidazol-3-ium bromide (2). White solid, yield 100%, mp 98–102°C. IR spectrum, v, cm⁻¹: 3432 (NH₂). ¹H NMR spectrum, δ , ppm: 7.44 d (1H, J = 1.8 Hz, =CH), 7.36 d (1H, J =1.8 Hz, =CH), 4.11 t (1H, J = 5.8 Hz, CH₂), 3.74 s (3H, CH₃), 2.98 t (1H, J = 5.8 Hz, CH₂), 2.58 s (3H, CH₃). ¹³C NMR spectrum, $\delta_{\rm C}$, ppm: 146.49, 123.76, 122.32, 51.53, 42.05, 35.65, 10.34. Found, %: C 35.19; H 5.73; N 17.61. C₇H₁₄BrN₃. Calculated, %: C 38.20; H 6.41; N 19.09.

Compound 5 was synthesized according to the procedure used for 2.

1-(2-Aminoethyl)pyridin-1-ium bromide (5). Yellow viscous liquid, yield 40%. IR spectrum, v, cm⁻¹: 3442 (NH₂). ¹H NMR spectrum, δ, ppm: 9.04 d (2H, J = 5.8 Hz, H_{Py}), 8.63 t (1H, J = 7.8 Hz, H_{py}), 8.14 t (2H, J = 7.1 Hz, H_{py}), 4.72 t (2H, J = 5.9 Hz, CH₂–C), 3.26 t (2H, J = 5.9 Hz, CH₂–C). ¹³C NMR spectrum, δ_C, ppm: 146.89, 146.26, 129.27, 64.71, 43.27. Found, %: C 39.32; H 6.25; N 12.22. C₇H₁₁BrN₂. Calculated, %: C 41.40; H 5.46; N 13.79.

3-(2-Aminoethyl)-1,2-dimethyl-1*H***-imidazol-3-ium hexafluorophosphate(V) (3a).** A mixture of compound **2** (5 mmol) with anhydrous methanol (10 mL) was stirred for 5 min, then potassium hexafluorophosphate was added and the mixture was stirred for about 2 h. Methanol was removed under vacuum, and acetonitrile (5 mL) was added to the residue. The suspension was stirred for 2 h and then filtered through celite. The filtrate was concentrated under vacuum to give pure compound **3a**. Yellow solid, yield 100%, mp 38–46°C. IR spectrum, v, cm⁻¹: 3650, 3640 (NH₂). ¹H NMR spectrum, δ , ppm: 7.46 d (1H, *J* = 1.9 Hz, =CH), 7.44 d (1H, *J* = 1.9 Hz, =CH), 4.17 t (2H, *J* = 6.1 Hz, CH₂), 3.80 s (3H, CH₃), 3.04 t (2H, *J* = 6.1 Hz, CH₂), 2.62 s (3H, CH₃). ¹³C NMR spectrum, δ_{C} , ppm: 146.43, 123.68, 122.16, 51.36, 42.00, 35.36, 9.59; ¹⁹F NMR spectrum, δ_{F} , ppm: –73.82, –75.32. Found, %: C 27.81; H 4.71; N 13.60. C₇H₁₄F₆N₃P. Calculated, %: C 29.48; H 4.95; N 14.74.

Compound 6 was synthesized according to the procedure used for **3a**.

1-(2-Aminoethyl)pyridin-1-ium hexafluorophosphate (6). White solid, yield 100%, mp 70–76°C. IR spectrum, v, cm⁻¹: 3415 (NH₂). ¹H NMR spectrum, δ, ppm: 8.91 d (2H, J = 5.8 Hz, H_{Py}), 8.59 t (1H, J = 7.9 Hz, H_{Py}), 8.10 t (2H, J = 7.9 Hz, H_{Py}), 4.62 t (2H, J = 5.5 Hz, CH₂–C), 3.22 t (2H, J = 5.3 Hz, CH₂–C). ¹³C NMR spectrum, δ_C, ppm: 146.65, 145.86, 128.83, 65.24, 42.89. ¹⁹F NMR spectrum, δ_F, ppm: –72.00, –73.50. Found, %: C 31.05; H 4.06; N 9.70. C₇H₁₁F₆N₂P. Calculated, %: C 31.36; H 4.14; N 10.45.

3-(2-Aminoethyl)-1,2-dimethyl-1H-imidazol-3-ium tetrafluoroborate (3b). A mixture of ionic compound 2 (5 mmol) with anhydrous methanol (10 mL) was stirred for 10 min, then silver tetrafluoroborate (5 mmol) was added and a pale yellow precipitate formed immediately. After 5 min of stirring the mixture was quickly filtered through celite which was washed with methanol. The filtrate was concentrated to give product 3b. Yellow viscous liquid, yield 100%. IR spectrum, v, cm⁻¹: 3450 (NH₂). ¹H NMR spectrum, δ , ppm: 7.30 d (1H, J=2.0 Hz, =CH), 7.27 d (1H, J = 2.0 Hz, =CH), 4.08 t (2H, J = 6.0 Hz, CH₂), 3.71 s (3H, CH₃), 3.02 t (2H, J = 6.0 Hz, CH₂), 2.53 s (3H, CH₃). ¹³C NMR spectrum, δ_C , ppm: 146.39, 123.60, 122.13, 51.49, 42.08, 35.36, 9.61. ¹⁹F NMR spectrum, δ_F, ppm: -151.63, -151.69. Found, %: C 36.39; H 5.89; N 18.78. C₇H₁₄BF₄N₃. Calculated, %: C 37.04; H 6.22; N 18.51.

Synthesis of compound 9a, 9b and 11. Ionic compound 3a (2 mmol) and anhydrous acetonitrile (10 mL) were stirred for 2 min, then hexafluoroglutaric anhydride 7 (2 mmol) was added dropwise. The reaction mixture was stirred for 5 min and silver oxide (1.5 mmol) was added to the solution in one portion. After the suspension was stirred for 15 min, it was filtered through celite which was then washed 3 times with acetonitrile. The combined filtrates were concentrated under reduced pressure to give product 9a.

[(5-{[(1,2-Dimethyl-1*H*-imidazol-3-ium-3-yl)methyl]amino}-2,2,3,3,4,4-hexafluoro-5-oxpentanoyl)oxy]silver hexafluorophosphate (9a). Yellow solid, yield 96%, mp 106–114°C. IR spectrum, v, cm⁻¹: 3420 (NH), 1712, 1689 (C=O). ¹H NMR spectrum, δ , ppm: 7.29 d (1H, *J* = 2.0 Hz, =CH), 7.24 d (1H, *J* = 2.0 Hz, =CH), 4.03 t (2H, *J* = 6.0 Hz, CH₂), 3.70 s (3H, CH₃), 2.98 t (2H, *J* = 6.0 Hz, CH₂), 2.53 s (3H, CH₃). ¹³C NMR spectrum, $\delta_{\rm C}$, ppm: 159.83, 158.73, 144.80, 122.31, 121.32, 118.12, 46.47, 34.63, 9.08. ¹⁹F NMR spectrum, $\delta_{\rm F}$, ppm: -73.93, -75.43, -117.37 t (*J* = 8.7 Hz), -120.39 t (*J* = 8.6 Hz), -125.18. Found, %: C 18.76; H 3.41; N 6.87. C₁₂H₁₃AgF₁₂N₃O₃P. Calculated, %: C 23.47; H 2.13; N 6.84.

Compounds **9b** and **11** were synthesized according to the same procedure used for **9a**.

[(5-{[(1,2-Dimethyl-1*H*-imidazol-3-ium-3-yl)methyl]amino}-2,2,3,3,4,4-hexafluoro-5-oxpentanoyl)oxy]silver tetrafluoroborate (9b). Yellow solid, yield 89%, mp 68–76°C. IR spectrum, v, cm⁻¹: 3414 (NH), 1710, 1686 (C=O). ¹H NMR spectrum, δ, ppm: 7.44 d (1H, J = 1.8 Hz, =CH), 7.36 d (1H, J = 1.8 Hz, =CH), 4.12 t (2H, J = 5.9 Hz, CH₂), 3.75 s (3H, CH₃), 2.98 t (2H, J = 5.9 Hz, CH₃), 2.58 s (3H, CH₃). ¹³C NMR spectrum, δ_C, ppm: 161.86, 160.64, 146.18, 123.33, 122.39, 47.76, 39.75, 35.78, 10.09. ¹⁹F NMR spectrum, δ_F, ppm: –115.89, –118.98, –125.95, –151.65, –151.70. Found, %: C 24.22; H 2.59; N 5.60. C₁₂H₁₃AgBF₁₀N₃O₃. Calculated, %: C 25.93; H 2.36; N 7.56.

[(2,2,3,3,4,4-Hexafluoro-5-oxo-5-{[2-(pyrdin-1ium-1-yl)ethyl]amino}pentanoyl)oxy]silver hexafluorophosphate (11). Yellow viscous liquid, yield 97%. IR spectrum, v, cm⁻¹: 3417 (NH), 1709, 1686 (C=O). ¹H NMR spectrum, δ, ppm: 8.97 s (1H, NH), 8.91 d (2H, J = 6.0 Hz, H_{Py}), 8.50 t (1H, J = 7.3 Hz, H_{Py}), 8.02 t (2H, J = 7.3 Hz, H_{Py}), 4.76 t (2H, J = 5.3 Hz, CH₂-C), 3.88 t (2H, J = 5.3 Hz, CH₂-C). ¹³C NMR spectrum, δ_C, ppm: 161.90, 160.81, 147.04, 146.29, 129.30, 61.36, 41.00. ¹⁹F NMR spectrum, δ_F, ppm: -72.11, -73.61, -115.92 t (J =5.0 Hz), -118.89 t (J = 5.0 Hz), -125.99. Found, %: C 23.91; H 2.44; N 4.22. C₁₂H₁₀AgF₁₂N₂O₃P. Calculated, %: C 24.14; H 1.69; N 4.69.

3-{2-[(4-Carboxy-2,2,3,3,4,4-hexafluorobutanoy])oxy]ethyl}-1,2-dimethyl-1*H***-imidazol-3-ium hexafluorophosphate (13). Ionic liquid 12 (1 mmol) and anhydrous THF (1 mL) were stirred for 5 min, then hexafluoroglutaric anhydride 7 (1.1 mmol) was added dropwise, and the solution was stirred at 28°C for 3 h. The solvent was removed under vacuum to give compound**

13. White solid, yield 98%, mp 50–54°C. IR spectrum, v, cm⁻¹: 3163 (OH), 1784, 1686 (C=O). ¹H NMR spectrum, δ, ppm: 8.22 s (1H, OH), 7.28 d (2H, J = 6.6 Hz, =CH), 4.63 t (2H, J = 5.0 Hz, CH₂–C), 4.46 t (2H, J = 4.46 Hz, CH₂–C), 3.71 s (3H, CH₃), 2.53 s (3H, CH₃). ¹³C NMR spectrum, δ_C, ppm: 159.70, 158.86, 146.47, 123.71, 122.33, 67.17, 47.18, 35.97, 10.19. ¹⁹F NMR spectrum, δ_F, ppm: -72.14, -73.64, -119.16 t (J = 10.0 Hz), -119.52 t (J = 9.2 Hz), -124.84. Found, %: C 29.14; H 3.69; N 5.60. C₁₂H₁₃F₁₂N₂O₄P. Calculated, %: C 28.36; H 2.58; N 5.51.

({5-[2-(1,2-Dimethyl-1*H*-imidazol-3-ium-3-yl)ethoxy]-2,2,3,3,4,4-hexafluoro-5-oxopentanoyl}oxy)silver hexafluorophosphate (14). A mixture of carboxylic acid 13 (1 mmol) with anhydrous acetonitrile (1 mL) was cooled down to 0°C and stirred for 5 min. Ag₂O (0.5 mmol) was added in one portion, and the mixture was stirred for 15 min. The precipitate was concentrated by centrifugation and washed with acetonitrile 3 times. The combined organic fractions were concentrated under vacuum. Yellow solid, yield 87%, mp 58–62°C. IR spectrum, v, cm⁻¹: 1784, 1686 (C=O). ¹H NMR spectrum, δ , ppm: 7.39 d (1H, J = 1.8 Hz, =CH), 7.23 d (1H, J=1.7 Hz, =CH), 4.55 m (2H, CH₂-C), 4.41 m (2H, CH₂–C), 3.69 s (3H, CH₃), 2.51 s (3H, CH₃). ¹³C NMR spectrum, $\delta_{\rm C}$, ppm: 161.69, 159.58, 146.44, 123.40, 122.41, 65.99, 47.38, 35.88, 10.22. ¹⁹F NMR spectrum, $\delta_{\rm F}$, ppm: -72.08, -73.58, -115.91 t (J= 10.0 Hz), -118.03 t (J = 10.0 Hz), -119.16. Found, %: C 26.11; H 2.72; N 5.38. C₁₂H₁₂AgF₁₂N₂O₄P. Calculated, %: C 23.43; H 1.97; N 4.55.

Synthesis of ruthenium complex 15. A mixture of G–H 2nd complex (1.0 eq, 15.7 mg, 0.025 mmol) with anhydrous THF (1 mL) was stirred for 5 min, then an appropriate silver carboxylate (**9a**, **9b** or **11**) (1.0 eq, 0.025 mmol) mixed with 0.2 mL of anhydrous acetonitrile, was added dropwise. The vessel containing silver salt was washed with THF (30.3 mL), and the content was added to the reaction mixture. The reaction mixture was stirred at 50°C for 3 h. The white precipitate (silver chloride) was filtered off, and the filtrate was concentrated under reduced pressure to give compound **15**.

Cross olefin metathesis reaction catalysis by compound 15. In a glove box, the catalyst **15** (1 mol %) and toluene (1 mL) were mixed and stirred for 2 min. Then allylbenzene (0.5 mmol, 59.1 mg) was added. After stirring for 15 min, methyl acrylate (0.5 mmol, 43 mg) was added to the system. The mixture was heated up to 50°C and stirred for 1 h, then concentrated under reduced pressure and purified by silica gel chromatography (ethyl acetate/petroleum ether = 3 : 97).

CONCLUSIONS

Analytically pure amido-functionalized ionic liquids have been synthesized and used in reaction with hexafluoroglutaric anhydride giving ion-functionalized carboxylic acids. The acids have been reacted with Ag_2O to produce silver carboxylates in high yields. The newly formed G–H 2nd type catalysts with one chlorine ion replaced by ion-functionalized carboxylate have been evaluated in cross olefin metathesis reaction. The new G–H 2nd complexes have not demonstrated improvement in their activity and E/Z selectivity in comparison with the parent G–H 2nd catalyst.

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CONFLICT OF INTEREST

No conflict of interest was declared by the authors.

SUPPLEMENTARY MATERIALS

Supplementary materials are available for this article at https://doi.org/10.1134/S1070363220110237 and are accessible for authorized users.

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