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Iridium-Catalyzed Selective Cross-Coupling of Ethylene Glycol and Methanol to Lactic Acid

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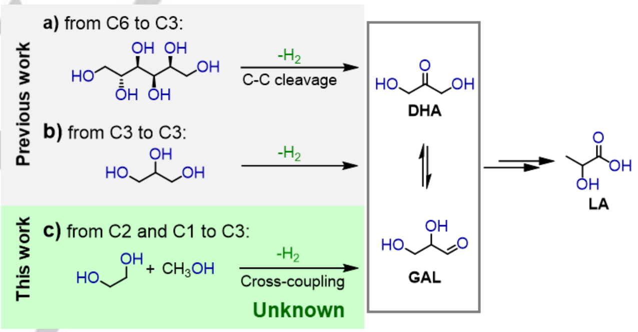
Abstract: We report an atom-economic approach that has an unprecedentedly high selectivity for lactic acid (LA) synthesis based on a catalytic dehydrogenative cross-coupling using cheap and bulk ethylene glycol and methanol. Our method relies on the synthesis and utilization of a novel iridium catalyst bearing three N-heterocyclic carbenes derived from 1.3-dimethylbenzimidazolium salts, which exhibits an outstanding activity in producing LA (turnover frequency (TOF) up to 3660 h^{-1}) due to an elegant metal-ligand cooperation.

Lactic acid (LA) and its derivatives are important platform molecules,[1-2] and have attracted extensive attention due to their broad applications with projected market demand expected to reach 4×10^5 tons per annum by 2022.[1] However, the global production capacity of LA is inadequate, owing to limitations with conventional chemocatalysis- and fermentation-based process.[2] Recently, two catalytic dehydrogenation approaches have been developed for LA production either from sorbitol (C6, Figure 1a)[3] or from glycerol (C3, Figure 1b),[4] which are environmentally friendly and economically appealing.[5,6] It was commonly regarded dihydroxyacetone (DHA) and glyceraldehyde (GAL) as the key intermediates in both transformations (Figure 1).[3,4] However, the major side products, included ethylene glycol (EGO), methanol (MeOH), glycolaldehyde, and formaldehyde from the unwanted C-C cleavage of GAL, were the main negative issues for achieving high selectivity for these transformations.[4a]

In consideration that hydrogen-borrowing reactions have exhibited a great potential for the construction of C-C bonds,[5,7] particularly, in the methylation of alcohols,[8] the cross-coupling between EGO and MeOH (from C2+C1 to C3, Figure 1c) should theoretically be possible. This new protocol shall not only provide a straightforward and atom-economic synthetic route for LA production, but also inhibit the side reactions in the dehydrogenation of sorbitol and glycerol so as to further increase the selectivity and yield of LA. In addition, EGO and MeOH both are inexpensive, renewable and bulky alcohols. Their global production capacities in 2016 are 34 and 80 million tons, respectively,[8a,9] constituting appealing coupling partners for the hydrogen-borrowing cross-coupling reactions.



✓ An unprecedented atomic-economy strategy for LA synthesis;
✓ Using cheap biomass-derived alcohol feedstock;
✓ Highly selective C-C cross-coupling of simple alcohols.

Figure 1. Catalytic transformation approaches for LA synthesis.

However, the direct catalytical cross-coupling between EGO and MeOH is challenging,[10,11] as the viable catalyst has to be bifunctional, which should not only selectively dehydrogenate MeOH and EGO to formaldehyde and glycolaldehyde simultaneously, but also selectively accelerate the aldol-type cross-coupling to generate GAL rather than homo-coupling of aldehydes or overoxidation, esterification, or polymerization of EGO itself. Therefore, a metal-ligand cooperativity is crucial for the hypothesized transformation. Herein, we realized the first selective protocol for LA synthesis *via* dehydrogenative cross-coupling between EGO and MeOH (Figure 1c), where all possible byproducts are significantly suppressed. This is achieved by the utilization of a novel highly active iridium (Ir) catalyst that bears three N-heterocyclic carbenes (NHCs) derived from BenIm (*i.e.*, 1.3-dimethylbenzimidazolium) salts. This tris-NHC-Ir complex exhibits excellent catalytic activity and selectivity, with a turnover frequency (TOF) up to 3660 h^{-1}. The ligand effects can facilitate the electron removal from the Ir(I) center, along with the dehydrogenation of MeOH and EGO simultaneously, such that the Ir(I) center is readily oxidized to yield an active Ir(III) species in the catalytic cycle.

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NHC-Ir complexes have been reported and acted as bifunctional catalysts in the dehydrogenation of polyols[4a,c] and other reactions.[12] A mononuclear Ir dihydride complex that bears three NHCs was once isolated (in low yields; ~5-10%) from the C3 to C3 transformation mixture under Crabtree's standard reaction conditions.[4a] Unfortunately, this tris-NHC-Ir compound decomposed before its catalytic potential could be assessed, and subsequent attempts to isolate it again proved unsuccessful.[4a] As a strong σ-donor and a weak π-acceptor, NHC is widely used to tune the activity and the selectivity in various reactions.[12a-c] Here, we postulated that increasing the π-ring size and/or the number of NHCs within the coordination sphere of the Ir complex may enhance the steric bulkiness and the electron density of the Ir center, which may facilitate the formation and the catalytic function of the active tris-NHC-Ir compound.

Accordingly, a series of NHC-Ir complexes, bearing one to three NHCs, were synthesized using 1,3-dimethylimidazolium (**S1**: Im) or 1,3-dimethylbenzimidazolium (**S2**: BenIm) iodide (Figure 2). The mono-NHC-Ir(I) complexes (**1a**-**b**) were prepared from **S2** and $[Ir(COD)Cl]_2$ (COD = 1,5-cyclooctadiene) with KO^{t}Bu (^{t}Bu = *tert*-butyl) in good yields (~90%, Figure 2a).[12a] After the ligand exchange with CO, complexes **1c**-**d** were obtained in 89-93% yields. The bis-NHC-Ir(I) complexes (**2aa**, **2ba**, and **2bb**) were synthesized in moderate yields using $[Ir(COD)Cl]_2$ and the corresponding salts (**S1** or **S2**) with NaH as the base (Figure 2b). Subsequent ligand exchange gave complexes **2ab**, **2bc**, and **2bd** in excellent yields (95-96%). With $Ir(acac)(CO)_2$ (acac = acetylacetonato), tris-NHC-Ir(I) complex **3b** was successfully synthesized (Figure 2c), while the tris-NHC-Ir(III) complex **3a-2H** with two hydrides was isolated directly rather than the anticipated tris-NHC-Ir(I) complex **3a**. Importantly, complexes **3a-2H** and **3b** were stable in their solid-state (shelf-life more than 3 months at 25 ºC), and relatively stable for a couple of days in solution (*e.g.*, MeOH and dichloromethane). Gratifyingly, the dihydride complex **3b-2H** was readily prepared from **3b** in a moderate yield (>48%) by treatment with excess $NaBH_4$ at 25 ºC in *i*-PrOH.[12a]

Subsequently, detailed structures of **2bd** and **3b** were determined by single-crystal X-ray crystallography (Figure S39, Tables S3 and S4). The complex **2bd** contains one Ir(I) center with two NHCs and two CO ligands (Table S3), adopting a *cis* square planar geometry that is similar to **2ab**.[6a] Crystal structure analysis of complex **3b** (Figure S39, Table S4) clearly showed that the Ir(I) center connects to three NHCs and one CO ligand, again forming a square planar geometry. The average $Ir{-}C_{NHC}$ distance of **3b** is 2.059(6) Å, which is slightly shorter than those of **2bd** (2.06(8) Å), indicating stronger $Ir{-}C_{NHC}$ interactions in **3b**.

Density functional theory (DFT) calculations were performed to elucidate the electronic nature of the Ir carbonyl complexes bearing different number of NHCs. The calculations showed that the ionization potentials (IPs, in eV) anti-correlate to the number of NHCs. IPs follow the trend of $[Ir(Im)(CO)_3]^+$ (12.4) > $[Ir(Im)_2(CO)_2]^+$ (11.0) > $[Ir(Im)_3(CO)]^+$ (9.7), which can be ascribed to the strong σ-donating and weak π-accepting characters of the NHCs. Additionally, IPs can be further reduced by expanding the π-conjugation of NHC: $[Ir(BenIm)(CO)_3]^+$ (11.7) > $[Ir(BenIm)_2(CO)_2]^+$ (10.5) > $[Ir(BenIm)_3(CO)]^+$ (9.6). As we expected, all these results reveal that ligand effects can facilitate the electron removal from the initial complexes, leading to an easy oxidation of the Ir(I) to yield the desired Ir(III) species.

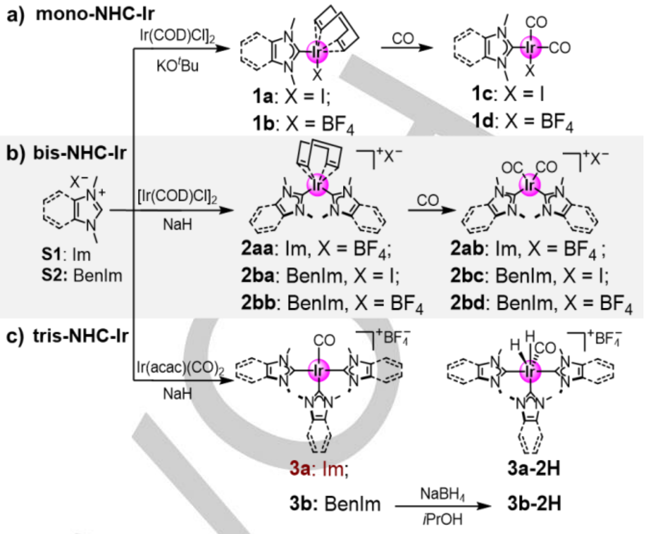

Figure 2. Syntheses of a) mono-NHC-Ir(I) complexes **1a-d**, b) bis-NHC-Ir(I) complexes **2aa-bd**, and c) tris-NHC-Ir complexes **3a-2H**, **3b**, **3b-2H** (**3a** was not accessible).

We then examined the feasibility of the dehydrogenative cross-coupling of EGO and MeOH to LA using tris-NHC-Ir(I) complex **3b** as the catalyst. The reaction was performed at 140 °C in the presence of 500 ppm **3b**, with 5 mmol EGO and 1.5 equiv. $Ba(OH)_2{\cdot}8H_2O$, in large excess of MeOH (2 mL). LA was formed within one hour in a quantitative yield (based on EGO) with an excellent selectivity (Figure 3), showing a high catalytic activity of **3b**. Encouraged by this excellent result, other reaction parameters including solvent, base, and reaction temperature were further surveyed (Table S1). Note that the yield and selectivity of LA decreased dramatically when using protic solvents like water, ethanol, or other primary alcohols; a similar decrease was observed in a solvent-free case (Table S1, entries 1-6). To highlight the crucial role of the MeOH, we found that all reactions with alternative solvent systems produced polyethylene glycol as the major byproduct. Moreover, decreasing the amount of MeOH from 10 to 5 or 2 equiv. resulted in inferior yields (>99% vs. 83% or 57%, Table S1, entries 7-8).

The base, $Ba(OH)_2$, also had a substantial impact on the transformation. Alkali metal hydroxides including LiOH, NaOH, and KOH all exhibited lower activities (Table S1, entries 9–11), whereas no reaction occurred with calcium hydroxide, probably due to its poor solubility (Table S1, entry 12). When the amount of $Ba(OH)_2$ was decreased to 1.2 equiv., LA was still obtained in a quantitative yield. Further decreasing the amount of $Ba(OH)_2$ to 1.0 equiv. resulted in a slightly decreased yield (95%), along with co-generation of a minor amount of the byproduct glycolic acid (~3%, assessed via HPLC; Table S1, entries 13-14). Lower temperatures also led to inferior yields (Table S1, entries 15-16).

Under the optimized reaction conditions, other NHC-Ir complexes were also examined: the catalytic efficiency increased along the mono < bis < tris sequence and that **3b** exhibited a higher TOF (2000 h^{-1}) than its analogue **3a-2H** (1500 h^{-1}). These results were likely attributed to our hypothesis that increasing the number and the π-ring size of the NHCs could facilitate oxidation of the Ir(I) to the Ir(III) species and improve the catalytic performance. Furthermore, several viable Ir and Ru catalysts (**4-10**) that exhibited a good catalytic activity in several dehydrogenative[5d] and cross-coupling[13] reactions were also investigated (Figure 3). Among them, the pincer-type Ru-PNP complexes (**9** and **10**)[5a,13b] displayed good TOFs (around 900 h^{-1}), further attesting the superior catalytic activity of **3b** (TOF around 2000 h^{-1}). When the catalyst loading of **3b** was reduced to 250 ppm, a TOF value as high as 3540 h^{-1} was obtained (Figure 3), and an even slightly higher TOF of 3660 h^{-1} was observed with the solid **3b-2H**. Pleasingly, the reaction still proceeded smoothly with the catalyst loading as low as 25 ppm (Table S1, entry 18), producing LA in a quantitative yield with a turnover number (TON) up to 4×10^4 within 48 hours.

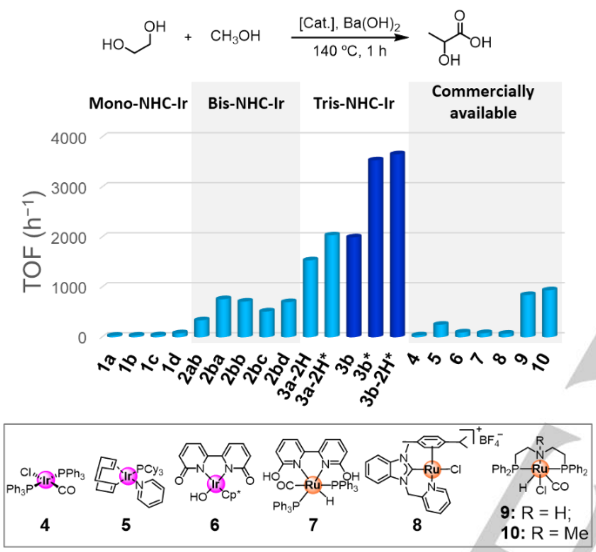

Figure 3. Catalytic activities of selected Ir and Ru complexes **1-10** in the dehydrogenative cross-coupling of EGO and MeOH to LA (With 5 mmol EGO, 500 ppm catalyst, 1.2 equiv. $Ba(OH)_2 \cdot 8H_2O$ and 2 mL MeOH at 140 °C for 1 h. Asterisks denote the catalyst loading at 250 ppm).

We subsequently performed several control experiments. The commercially available Ir/C and freshly prepared Ir nanoparticles exhibited much lower activities under the same reaction conditions (1-3%, Table S2, entries 1-2). Mercury poisoning experiments excluded the possibility of any nanoparticle participation (Table S2, entry 3).[14] In contrast, addition of excess PPh_3 significantly inhibited the reaction process (from 2000 h^{-1} to 134 h^{-1}, Table S2, entry 4), revealing that PPh_3 can replace NHCs. All these outcomes clearly suggested that complex **3b** behaved as a homogenous molecular catalyst in the reaction.

High-resolution mass spectroscopy (HR-MS) analysis of a dichloromethane extract of the reaction mixture (Figure 4a and S36) revealed that, either **3b** or **3b-2H** was added, we observed discrete signals for both **3b** (m/z: 659.23) and **3b-2H** (m/z: 661.23, Figure 4a). Besides, we also found an Ir complex $Ir(BenIm)_3^+$ **I** (m/z: 631.23, Figure 4a) and a peak at m/z 693.22 (Figure 4a), which was attributed as the dihydride NHC-Ir(III) complex **V**, as coordinated with glycolaldehyde. ^1H NMR spectroscopic studies were then carried out. When tris-NHC-Ir(III) **3a-2H** was heated in a mixture of CH_3OH/CD_3OD (1:1) with an excess amount of KOH at 60 °C for 1 hour (conditions similar to the cross-coupling reaction), two hydride signals appeared at −8.53 and −11.70 ppm, respectively (Figure 4b). The correlation of the two hydrides in two-dimensional correlation spectroscopy (2D-COSY) revealed that the hydrides adopt a *cis* configuration (Figure S30), similar to that previously reported for pincer complexes.[13b] Nearly identical hydride signals were detected in the ^1H NMR spectrum of tris-NHC-Ir(I) **3b** with KOH in a hot mixture of CH_3OH/CD_3OD (Figure 4b), implying that tris-NHC-Ir(I) **3b** could be converted into *cis*-dihydride species tris-NHC-Ir(III) **3b-2H**, which was further supported by a 2D-COSY study (Figure S31). When similar measurements were applied to **3b-2H** with excess $NaBH_4$ in *i*-PrOH (Figure 2c), ^1H NMR spectrum not only indicated the presence of the ***cis*-3b-2H** associated with peaks at δ −8.50 and −11.70 ppm, but also the *trans*-dihydride ***trans*-3b-2H** formation with a single peak at δ −7.93 ppm, guided by a similar peak reported in a prior study.[14]

A plausible mechanism for the dehydrogenative cross-coupling of glycol and MeOH to LA was sketched in Figure 4c. Initially, CO dissociated from tris-NHC-Ir(I) **3b** to form species **I** with a free coordination site.[15] Both EGO and MeOH were then dehydrogenated to give the corresponding aldehydes via hydrogen-borrowing pathway along with the formation of the dihydride Ir(III) species **VII** (Please see Figure S40 for more details and the related discussions). The aldehydes underwent a cross-aldol condensation to generate GAL, which further underwent dehydration to form pyruvaldehyde and converted to LA *via* an intramolecular Cannizzaro reaction under the high base conditions.[4f] DFT calculations were performed to estimate the free energy changes during the H_2 release and found that ligand effects apparently contribute to the catalyst regeneration to complete the catalytical cycle (Table S7).

Considering the excellent yield and selectivity observed in the tris-NHC-Ir-catalyzed dehydrogenative cross-coupling of EGO with MeOH, the applicability in other reported bio-polyols was also investigated. To our delight, when glycerol or sorbitol was applied instead of EGO, excellent outcomes were also attained under the otherwise identical reaction conditions. Remarkably, 86% conversion, 85% yield with >99% selectivity for LA could be achieved when sorbitol was dehydrogenated (one molecular sorbitol generates 2 equiv. LA), which is much better than the previous report (ca. 40% yield, one sorbitol for one LA production),[3] and further confirmed the metal-ligand cooperativity is of vital importance for this type of transformation.

In summary, we disclose an unprecedented dehydrogenative cross-coupling approach using the biomass feedstock EGO and MeOH to yield the platform molecule LA within one hour. The metal-ligand cooperativity is of vital importance, in which catalytic activity of the Ir(I) complexes increases in a sequence of mono-NHC-Ir < bis-NHC-Ir < tris-NHC-Ir. The tris-NHC-Ir complexes derived from benzimidazolium salts exhibits the best activity, with quantitative selectivity and TOF up to 3660 h^{-1}. The readily available raw substrates, a highly active catalyst, and clean hydrogen gas side product highlight the great potential of this new atom-economic and formerly quite challenging dehydrogenative C-C cross-coupling reaction, thereby opening up a new sustainable avenue for the synthesis of platform molecules.

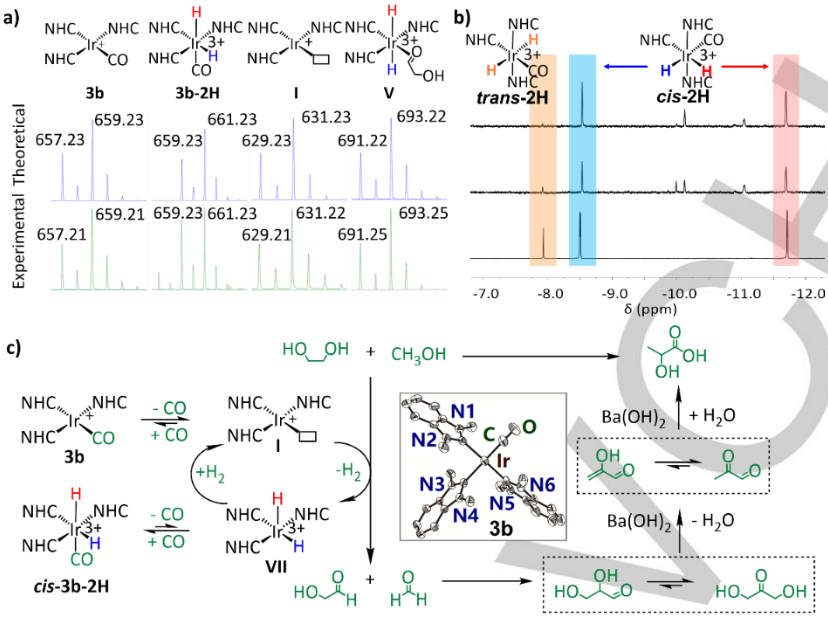

Figure 4. a) Theoretical (top) and experimental (bottom) high-resolution mass spectroscopy spectra. b) Partial ^{1}H NMR spectra of tris-NHC-Ir complexes **3a-2H** (top), **3b** (middle) with 10 equiv. KOH after heating and **3b-2H** (bottom) in CH_3OH and CD_3OD (1:1) mixture. c) A plausible mechanism (ORTEP diagram of complex **3b** is provided; hydrogen and BF_4^- anions have been omitted for clarity).

Acknowledgements

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Keywords: Lactic acid • cross-coupling • ethylene glycol • methanol • N-heterocyclic carbene iridium complex

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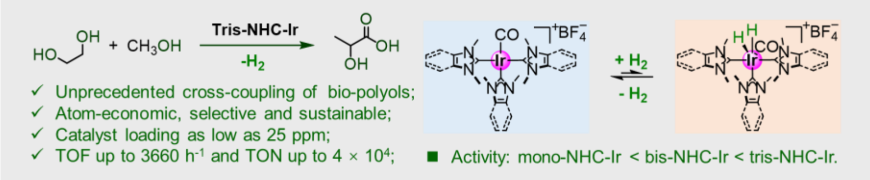

An unprecedented dehydrogenative C-C cross-coupling with cheap and bulky ethylene glycol and methanol has been developed by using a novel iridium catalyst bearing three N-heterocyclic carbenes, which presents as a highly selective and atom-economic approach for lactic acid synthesis (TOF up to 3660 h^{-1}).

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