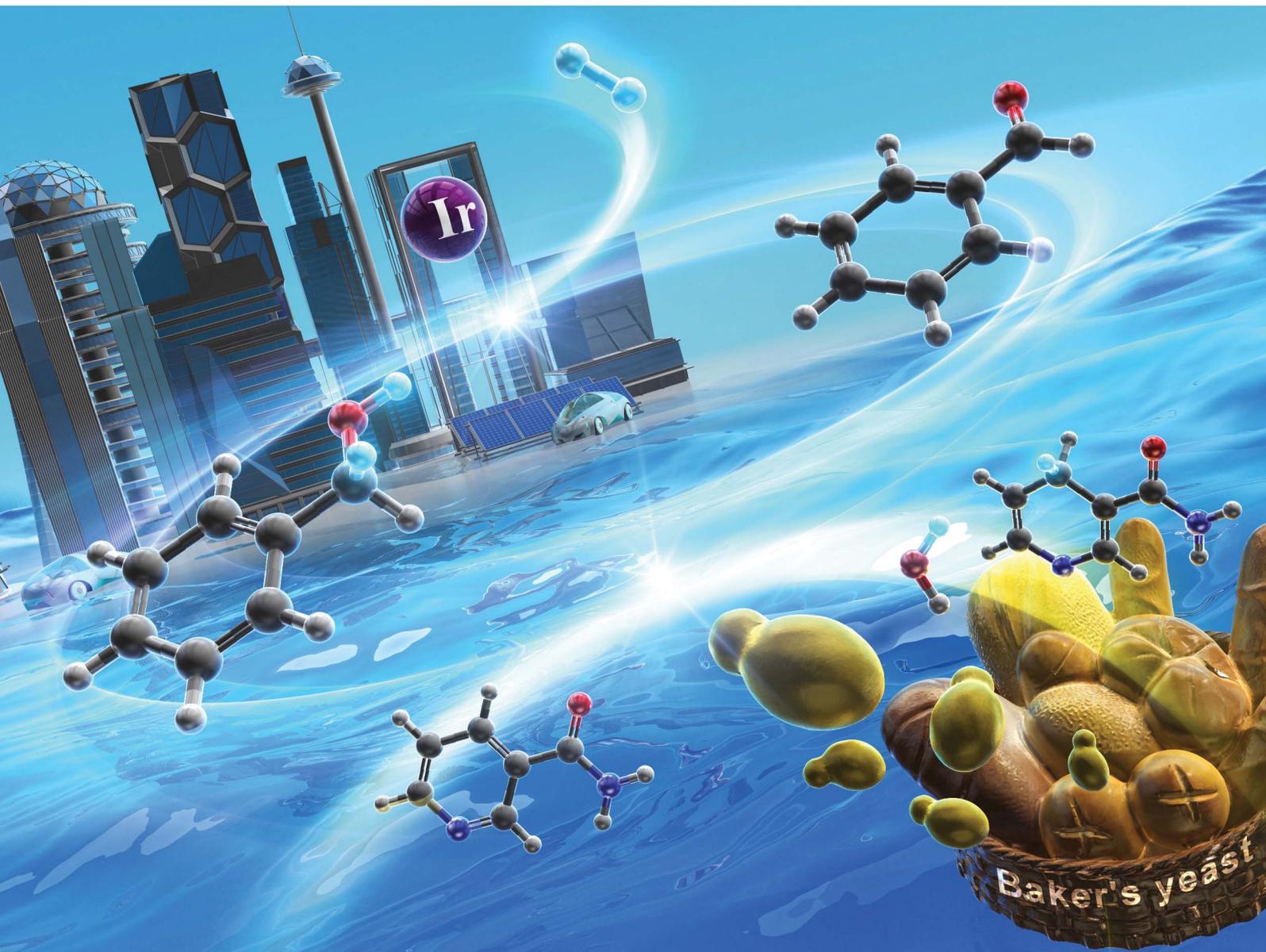


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Hydrogen gas production and storage cycle with benzyl alcohol/benzaldehyde

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Hydrogen gas (H₂) has attracted significant attention as a clean energy source. To realise a sustainable society, the development of methods for green H₂ production from water and safe H₂ storage is essential. Organic hydrides, which can store H₂ via covalent bonding under ambient conditions, are promising H₂ storage materials. Primary alcohols, which can be produced from biomass and release H₂ under mild conditions (80–220 °C) due to their small dehydrogenation enthalpies, have garnered attention as new candidates for application as organic hydrides. In this work, we demonstrated a reversible H₂ storage cycle with primary alcohols/aldehydes. Specifically, we focused on benzyl alcohol and relatively stable benzaldehyde. Benzyl alcohol was completely dehydrogenated by warming (180 or 220 °C) in the presence of a metal complex catalyst to obtain benzaldehyde, which was then completely hydrogenated to benzyl alcohol using the same catalyst under H₂ (1 atm), thereby accomplishing a reversible H₂ storage cycle. Additionally, we used alcoholic fermentation with baker's yeast as a hydrogenation method without requiring precious metal catalysts or H₂, and benzaldehyde was completely hydrogenated to store hydrogen directly from water and nicotinamide adenine dinucleotide (NADH). This work revealed the reversible H₂ storage capability of primary alcohols/aldehydes as organic hydrides, and conceptually demonstrated a green H₂ production and storage cycle by combining hydrogen storage using alcoholic fermentation and H₂ release.

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Introduction

Hydrogen gas (H₂) is indispensable for industries (e.g., petroleum refining,¹ steel manufacturing,² and ammonia synthesis³). Additionally, H₂ has attracted attention as a clean energy source alternative to fossil fuels⁴ due to its high gravimetric energy density (140.4 MJ kg⁻¹),⁵ which is higher than that of coke (28.8 MJ kg⁻¹) and gasoline (40.0 MJ kg⁻¹), and its combustion (H₂ + (1/2)O₂ = H₂O) is not accompanied by CO₂ emissions.⁶ However, current H₂ production methods mainly use fossil fuels, which in turn lead to CO₂ emissions.^{7–9} Additionally, current H₂ storage and transportation methods, such as compressed H₂ (300–700 bar) and liquid hydrogen (–253 °C), require high pressure or cryogenic temperature and face safety risks (e.g., flammability and explosiveness).^{10,11} Therefore, for the practical use of hydrogen energy, the development of efficient methods for green H₂ production from water and for safe H₂ storage and transportation is required.¹²

Porous materials (80 bar, –200 °C), hydrogen storage alloys (1–8 bar, –40–20 °C), and organic hydrides have been mainly reported as hydrogen storage materials.^{13,14} Among these, organic hydrides, which can store H₂ reversibly via covalent bonding under ambient conditions, have attracted attention as safe H₂ storage materials. Additionally, most organic hydrides exist as liquids under ambient conditions and are expected to enable hydrogen storage and transport by using the existing infrastructure for gasoline.¹⁵ From these advantages, organic hydrides are one of the promising hydrogen storage materials that enable relatively cost-effective and large-scale storage and transportation of hydrogen.¹⁶ However, most typical organic hydrides (e.g., toluene/methylcyclohexane) are produced from fossil resources,¹⁷ and they require high temperature conditions (e.g., >250 °C) for H₂ release due to their large dehydrogenation enthalpies.^{18,19} These high temperature conditions consume 25–30% of the energy obtained from the released H₂.²⁰ Nitrogen-containing heterocyclic compounds have attracted attention due to their small dehydrogenation enthalpies, but their high melting points (e.g., *N*-ethylcarbazole: 69 °C)²¹ make them difficult to transport using existing gasoline infrastructure.²² Based on the above situation, the use of organic compounds, which have small dehydrogenation enthalpies and exist as liquids under ambient conditions, is desirable.²⁰

As shown in Table 1, alcohols, which have small dehydrogenation enthalpies, can release H₂ under milder conditions

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Table 1 Characteristics of organic hydrides

Entry	Organic hydride	Dehydrogenation enthalpy (kJ mol ⁻¹)	Hydrogen storage density (wt%)	Liquid range (°C) (H ₂ adduct/H ₂ acceptor)	Ref.
1		205 (68 per H ₂)	6.2	-127–101/–95–111	23
2		585 (65 per H ₂)	6.2	-58–371/–34–398	23
3		300 (50 per H ₂)	5.2	<20–280/68–270	23
4 (This work)		54	1.9	-15–205/–56–179	24
5		68	4.4	-114–79/–123–20	25
6		—	6.5	-13–195/15–51	—
7		—	<5.6	146–NA/NA–NA	—

(80–220 °C)^{26–33} than those of typical organic hydrides and often exist as liquids under ambient conditions. Therefore, alcohols have recently attracted attention as new candidates for organic hydrides.³⁴ Among them, primary alcohols can be easily produced from biomass^{35,36} and are expected to be used as sustainable and environmentally friendly organic hydrides.

In this work, we achieved reversible H₂ storage using primary alcohols/aldehydes *via* hydrogenation and dehydrogenation reactions under mild conditions. Specifically, to conceptually demonstrate a reversible H₂ storage cycle with primary alcohols/aldehydes, we focused on benzyl alcohol (H₂ adduct), which can be produced from biomass,³⁷ and benzaldehyde (H₂ acceptor), which is relatively stable.³⁸ Benzyl alcohol was completely dehydrogenated to benzaldehyde by simply warming it with an iridium catalyst.³⁹ Subsequently, benzaldehyde was completely hydrogenated to benzyl alcohol with the same iridium catalyst under H₂ (1 atm), thereby accomplishing a reversible H₂ storage

cycle with benzyl alcohol/benzaldehyde. Additionally, we used alcoholic fermentation with baker's yeast²⁶ as a hydrogenation method without requiring precious metal catalysts or H₂, and benzaldehyde was completely hydrogenated to store hydrogen directly from water and nicotinamide adenine dinucleotide (NADH) contained in baker's yeast. As shown in Fig. 1, this work conceptually demonstrated a green H₂ production and storage cycle by combining hydrogen storage using alcoholic fermentation and H₂ release.

Results and discussion

Reversible H₂ storage cycle

In this work, we used aqua(2,2'-bipyridine-6,6'-dionato)(pentamethylcyclopentadienyl)iridium(III) (**Ir cat.**) as the hydrogenation and dehydrogenation catalyst.³⁹ **Ir cat.** is known for dehydrogenating alcohols under mild conditions (<250 °C) without using oxidants in various solvents,^{26,39} and has a high durability and long lifetime with a catalytic turnover number exceeding 100 000. In addition, **Ir cat.** enables the reversible transformation between secondary alcohols and ketones (*e.g.*, at least 8 cycles for 2-propanol/acetone³⁹), and the technology for recovering iridium is mature.⁴⁰ Therefore, **Ir cat.** also has reusability and recyclability. As shown in Fig. 1, benzyl alcohol was dehydrogenated with **Ir cat.** in *p*-xylene, which is often used in the dehydrogenation of alcohols by **Ir cat.**³⁹ From the perspective of sustainability, the use of *p*-xylene is undesirable in the future, and therefore, *p*-xylene was used only for the analysis and demonstration of the dehydrogenation and hydrogenation of benzyl alcohol/benzaldehyde. In other parts, from the perspective of sustainability, pinane was used as an environmentally friendly solvent alternative to *p*-xylene.

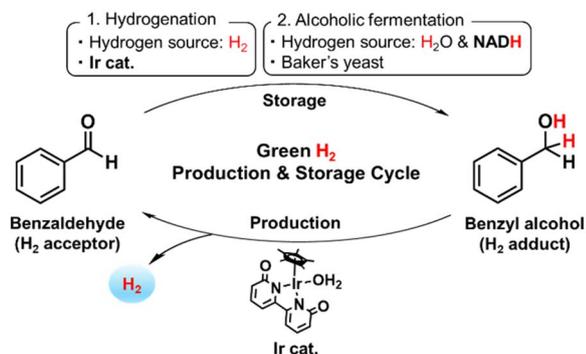
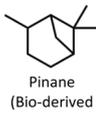


Fig. 1 H₂ production and storage cycle with benzyl alcohol/benzaldehyde.



Table 2 Dehydrogenation of benzyl alcohol in *p*-xylene or pinane with Ir cat.

Entry	Solvent	Temperature (°C)	Catalyst (mol%)	Reaction time (h)	Conversion ^a (%)
1	 <i>p</i> -Xylene	180 (Reflux)	0.5	10	100
2	 Pinane (Bio-derived solvent)	220 (Reflux)	1.5	4	98.0

^a Determined by ¹H NMR.

As shown in Table 2, Entry 1, and Fig. S1, benzyl alcohol was warmed in the presence of Ir cat. to be completely dehydrogenated to benzaldehyde. This full conversion was achieved by conducting the dehydrogenation reaction in an open system, where the evolved H₂ that can serve as a reactant for the hydrogenation reaction is continuously released outside the system to proceed the dehydrogenation reaction only. Additionally, as shown in Fig. S2, benzyl alcohol was dehydrogenated in a sealed system, and the evolved gas was analysed by a gas chromatography (GC) analysis. Even in a sealed system, where the evolved H₂ can remain, the endothermic nature of the dehydrogenation reaction can shift the equilibrium of the transformation between benzyl alcohol and benzaldehyde towards the production of benzaldehyde and H₂ at high temperatures. As shown in Fig. S3 and S4, the dehydrogenation of benzyl alcohol produced benzaldehyde and H₂ without any gaseous or liquid by-products. In addition, the amount of released H₂ corresponding to the conversion of benzyl alcohol to benzaldehyde determined by ¹H NMR spectroscopy was confirmed.

The dehydrogenation reaction of organic hydrides requires significant energy under high temperature conditions, making its kinetic analysis generally considered more important than that of the hydrogenation reaction.^{41,42} Therefore, as shown in Fig. 2, we conducted a kinetic analysis of the dehydrogenation reaction of benzyl alcohol with Ir cat. (details of the kinetic analysis and the reaction rate equations are shown in the SI). Based on the reaction rate constants summarised in Table S1

and the Arrhenius plot shown in Fig. 2b, the activation energy for the dehydrogenation of benzyl alcohol with Ir cat. was estimated to be +39.2 kJ mol⁻¹. This activation energy is lower than the standard reaction enthalpy for dehydrogenation of benzyl alcohol to benzaldehyde and H₂ ($\Delta H^\circ = +53.9$ kJ mol⁻¹).²⁴ This low activation energy is ascribed to the unique catalytic mechanism of Ir cat. (as shown in Fig. S5),⁴³ and is expected to reduce the energy consumption for H₂ release.

As mentioned in the aforementioned part, *p*-xylene is produced from fossil resources,⁴⁴ and therefore, the use of bio-derived solvents is desirable to develop sustainable H₂ release methods. In this work, we replaced *p*-xylene with pinane, which is a green solvent derived from pine tree oil.^{45–47} As shown in Table S2, the dehydrogenation of benzyl alcohol in pinane was revealed to proceed more slowly than that in *p*-xylene (Fig. S6), and increasing the amount of Ir cat. achieved reaction rates and conversion comparable to those in *p*-xylene (Table 2, Entry 2 and Fig. S7). In our ongoing work, our objective is to make the H₂ release method for primary alcohols more environmentally friendly by using non-precious, earth-abundant metal catalysts (e.g., manganese⁴⁸).

Subsequently, as shown in Fig. 1, benzaldehyde was hydrogenated with Ir cat. in *p*-xylene or pinane under H₂ (1 atm). As shown in Table 3 and Fig. S8 and S9, benzaldehyde was

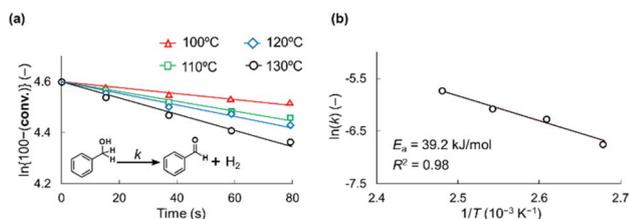


Fig. 2 (a) Dehydrogenation of benzyl alcohol with Ir cat. at 100–130 °C. (b) Arrhenius plots used to estimate the activation energy for the dehydrogenation of benzyl alcohol with Ir cat.

Table 3 Hydrogenation of benzaldehyde in *p*-xylene or pinane with H₂ and Ir cat. ^a

Entry	Solvent	Reaction time (h)	Conversion ^b (%)
1	 <i>p</i> -Xylene	6	100
2	 Pinane	4	100

^a Ir cat.: 0.5 mol% (vs. benzyl alcohol); temperature: 60 °C.^b Determined by ¹H NMR.

Table 4 Characteristics of various hydrogenation methods of organic hydrides

Entry	Hydrogenation method	Processes	Catalyst	Hydrogen source	Temperature (°C)	Ref.
1	Conventional H ₂ storage methods	Multiple processes	Precious metal catalyst	H ₂ (>30 bar)	>120	17
2	Electrochemical hydrogenation	Single process	Precious metal catalyst	H ₂ O	25	16
3	Alcoholic fermentation	Single process	Baker's yeast	H ₂ O and NADH	25	26

completely hydrogenated not only in *p*-xylene but also in pinane to produce benzyl alcohol and store H₂. From the above results of hydrogenation and dehydrogenation, in this work, a reversible H₂ storage cycle with benzyl alcohol/benzaldehyde is accomplished, and benzyl alcohol/benzaldehyde was conceptually demonstrated as an organic hydride (as shown in Fig. 1).

Green H₂ production & storage cycle

As shown in Table 4, Entry 1, conventional H₂ storage methods for organic hydrides require pure high-pressure H₂, and the entire process of H₂ production and storage (such as H₂ production *via* water electrolysis, purification, and the hydrogenation of organic hydrides with high-pressure H₂) consists of multiple steps and accompanied by energy consumption.^{49–51} Recently, as shown in Table 4, Entry 2, electrochemical hydrogenation has emerged as a simple method that can store hydrogen directly from water into organic hydrides without using H₂.¹⁶ However, even in the electrochemical hydrogenation, precious metal catalysts are often required. Therefore, from the perspective of sustainability, the development of hydrogen storage methods without requiring precious metal catalysts is desirable.

In this context, as shown in Table 4, Entry 3, alcoholic fermentation with baker's yeast can hydrogenate aldehydes to primary alcohols using water and nicotinamide adenine dinucleotide (**NADH**), which is a coenzyme contained in baker's yeast, as hydrogen sources.^{26,52} During alcoholic fermentation, **NADH** is oxidised to **NAD**⁺, but **NAD**⁺ can be regenerated to **NADH** by adding glucose.^{53,54} Therefore, **NADH** can be used as a renewable hydrogen source. In addition, alcoholic fermentation with baker's yeast can hydrogenate aldehydes under ambient conditions, which are milder than those of the conventional hydrogenation methods (*e.g.*, >120 °C). Furthermore, the hydrogen storage process based on alcoholic fermentation without using H₂ does not require the conventional multiple H₂ storage processes, which are energy-intensive. From the above, alcoholic fermentation with baker's yeast can be used as a novel environmentally friendly hydrogenation method for aldehydes, and the series of alcoholic fermentation and dehydrogenation processes is suggested to be more energy-efficient than that for conventional organic hydrides.

In this work, as shown in Fig. 1, we used alcoholic fermentation with baker's yeast as a hydrogenation method for benzaldehyde without requiring precious metal catalysts or H₂. Coexistence of benzaldehyde with baker's yeast (1 g) in water allowed benzaldehyde to be completely hydrogenated and to

Table 5 Hydrogenation of benzaldehyde in water with baker's yeast^a

Entry	Baker's yeast (g)	Reaction time (h)	Conversion ^b (%)
1	1.0	6	100
2	3.0	2	100

^a Temperature: 25 °C. ^b Determined by ¹H NMR.

store hydrogen directly from water and **NADH** after stirring for 6 h (Fig. S10 and Table 5, Entry 1). In addition, increasing the amount of baker's yeast to 3 g allowed the complete hydrogenation of benzaldehyde within 2 h (Fig. S11 and Table 5, Entry 2). These reaction times were comparable to or shorter than those required for the hydrogenation with **Ir cat.**, indicating that alcoholic fermentation with baker's yeast has the potential to store hydrogen with a time scale and efficiency comparable to the hydrogenation with **Ir cat.** In addition, baker's yeast can be easily separated from the produced alcohols,²⁶ enabling its combination with the subsequent dehydrogenation process without the deactivation of baker's yeast caused by the temperatures required for the dehydrogenation of alcohols. From the above results, we achieved direct hydrogen storage without requiring precious-metal catalysts or H₂ by using alcoholic fermentation. As shown in Fig. 1, a green H₂ production and storage cycle with benzyl alcohol/benzaldehyde was conceptually demonstrated by combining hydrogen storage using alcoholic fermentation and H₂ release.

Conclusions

This work achieved the dehydrogenation of primary alcohols (*e.g.*, benzyl alcohol) and hydrogenation of aldehydes (*e.g.*, benzaldehyde) under H₂ (1 atm) in pinane, a bio-derived solvent, and, for the first time, demonstrated that primary alcohols/aldehydes serve as organic hydrides. In addition, we used alcoholic fermentation with baker's yeast, in which water and **NADH** serve as the hydrogen sources, to achieve direct hydrogen storage without requiring precious metal catalysts or H₂. Furthermore, a green H₂ production and storage cycle with primary alcohols/aldehydes was conceptually demonstrated by combining alcoholic fermentation with the mild H₂ release method. This cycle using primary alcohols/aldehydes can contribute to the development of an H₂ release method under mild conditions and environmentally friendly methods for green H₂ production and storage. In addition, as shown in Table 1, Entries 6 and 7, the use of ideal polyhydric primary alcohols with high hydrogen storage densities should lead to the



development of promising organic hydrides with performance comparable to that of other representative candidates (e.g., methylcyclohexane/toluene), while having small dehydrogenation enthalpies. In our ongoing work, we comprehensively consider the characteristics (e.g., hydrogen storage density and volatility) for organic hydrides and aim to apply this green H₂ production and storage cycle to more ideal primary alcohols/aldehydes (e.g., ethylene glycol/glyoxal) and to biomass (e.g., open-chain form of glucose).

Author contributions

Tomoyuki Ito: methodology, formal analysis, data curation, investigation, validation, writing – original draft, writing – review & editing. Takumi Ichimura: methodology, formal analysis, data curation, investigation, writing – original draft, writing – review & editing, and funding acquisition. Hitoshi Kasai: writing – review & editing. Kouki Oka: conceptualization, methodology, formal analysis, data curation, investigation, project administration, supervision, writing – review & editing, funding acquisition, resources.

Conflicts of interest

The authors declare no conflicts of interest.

Data availability

The data supporting this article have been included as part of the supplementary information (SI). Supplementary information: materials and methods, ¹H NMR spectra, gas chromatograms, mechanism for the dehydrogenation of alcohols with Ir cat., and tables. See DOI: <https://doi.org/10.1039/d5se01721a>.

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