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Round and Round with Palladium

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Abstract: An introduction to catalytic cycles by using the Wacker process that converts ethene to ethanal is complemented by illustrating the working principles of a chemiresistor to detect ethene at the parts-per-billion level.

Keywords: Catalysis · Chemical education · Ethene · Sensor · Wacker process

After inital observations by Phillips in 1894,[1] the conversion of ethene to ethanal (acetaldehyde) catalysed by $PdCl_2$ in the presence of O_2 and a copper(II) salt was described in 1959.[2] This was developed industrially as the Wacker (or Wacker-Hoechst) process. The name 'Wacker' originates from the German chemical company Wacker Chemie AG, founded in 1914 by the businessman Alexander Wacker. The German chemical company Hoechst AG acquired a stake in the Wacker process through prudent patent applications.[3] The Wacker alkene oxidation has subsequently been advanced as a more general process for converting alkenes to organic carbonyl compounds, with some of the most recent work addressing improved control over product selectivity.[4,5] From a didactic point of view, the original Pd-catalysed ethene-to-ethanal conversion makes an excellent introduction to catalytic cycles, and I present this at an introductory level in the first section of this article. To provide additional interest for students, the second section of this column highlights an innovative application of the Wacker process to detect ethene at levels as low as 15 ppb. Such low detection limits are relevant to the agriculture and food industries – ethene is a plant hormone that has roles in plant development, blooming of flowers and fruit ripening. For example, the majority of fruits ripen when the concentration of ethene produced by the plant is in the range 0.1–1 ppm, while flowers begin to bloom with an ethene concentration <0.1 ppm.[6] Within the food industry, monitoring ethene production is important so as to prevent over-ripening and spoilage of fruit after harvesting.

Wacker Ethene Oxidation

The conversion of ethene to ethanal using Pd(II) as a catalyst in aqueous solution is represented in Eqn. 1. The key to making this an industrially viable catalytic reaction is the regeneration of Pd(II) using $CuCl_2$ (Eqn. 2) followed by rapid oxidation of CuCl to $CuCl_2$ (Eqn. 3). The overall reaction is given in Eqn. 4, and the cycle in Fig. 1 illustrates the Pd(II)-catalysed oxidation of ethene under aqueous acidic conditions.

$$C_2H_4 + H_2O + [Pd^{II}Cl_4]^{2-} \rightarrow CH_3CHO + Pd^0 + 2HCl + 2Cl^- \quad (1)$$
$$Pd^0 + 2CuCl_2 + 2Cl^- \rightarrow [Pd^{II}Cl_4]^{2-} + 2CuCl \quad (2)$$
$$2CuCl + 1/2O_2 + 2HCl \rightarrow 2CuCl_2 + H_2O \quad (3)$$
$$C_2H_4 + 1/2O_2 \rightarrow CH_3CHO \quad (4)$$

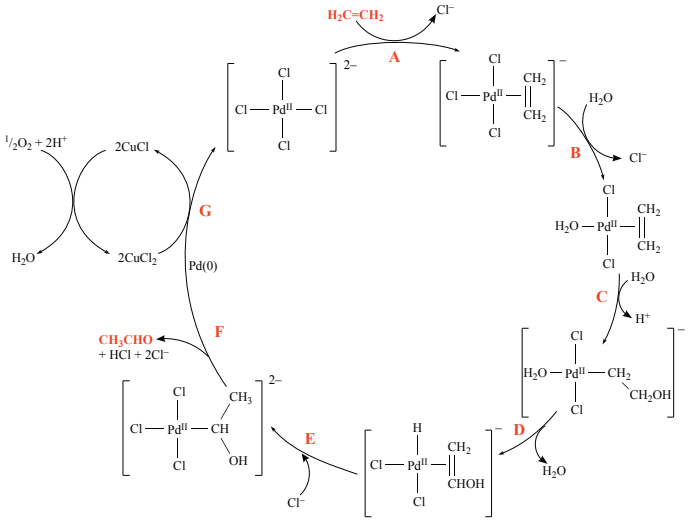

Fig. 1. A schematic representation of the catalytic cycle for the Wacker process.

Steps **A–G** in the catalytic cycle can be summarized as:
A ligand substitution (introduction of coordinated ethene);
B ligand substitution (H_2O displaces Cl^-);
C nucleophilic attack by H_2O on coordinated ethene;
D β-hydrogen elimination;
E H atom migration to give a σ-bonded $CH(OH)CH_3$ group;
F reductive elimination (Pd is reduced as ethanal is eliminated);
G regeneration of the Pd(II) catalyst.

Thus, the Wacker process can be used as a didactic tool to either introduce or reinforce a number of general organometallic reaction types, as well as different bonding modes (π-donor and π-acceptor ligands, σ- and π-bonded ligands). The IUPAC etanomenclature (η^2-ethene) can also be illustrated.

The Wacker process in the form of the more advanced Tsuji-Wacker alkene oxidation can be used to discuss the complications of using asymmetrical alkenes, $RHC{=}CH_2$. In step **C** in Fig. 1, nucleophilic attack may lead to Markovnikov or anti-Markovnikov addition and the subsequent formation of two regioisomers of the final organic carbonyl compound.[5] Grubbs observed that the use of a nitrite cocatalyst led to selective anti-Markovnikov alkene oxidation,[4] and this is of relevance to the next section in this article.

It is worth noting that although palladium-based catalysis for the conversion of ethene to ketones and aldehydes is the workhorse in industry, palladium-free catalysts are under development with the aim of enhancing efficiency, selectivity and sustainability of the processes.[7]

From Wacker to a Chemiresistive Sensor for Ethene

In this section, I take the Wacker process a step further with a topic that introduces sensors and carbon nanotubes. The struc-

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tures and properties of carbon nanotubes could be introduced at a relatively basic level[8] before their application in a sensor to detect ethene is described.

In the agricultural and food industries, levels of the plant hormone ethene have traditionally been measured using gas chromatography. Although a range of electrochemical and spectroscopic sensors has been developed,[9] their commercial application may be impeded by detection limits, speed of response and lack of a robust design. It is also necessary to be able to selectively detect ethene in the presence of other volatile organic compounds (VOCs).

In 2020, Swager and his team at Massachusetts Institute of Technology reported an ingeneous device[10,11] that integrates a simplified Wacker catalytic cycle within a chemiresistive sensor incorporating single-walled carbon nanotubes (SWCNTs).[12] Ethene is detected through its consumption in a Pd-catalysed Wacker-type process. Since SWCNTs are p-type semiconductors, the change between electron-rich Pd(0) (an n-dopant) and electron-poor Pd(II) (a p-dopant) during the catalytic cycle (Eqns. 1 and 2) reversibly alters the level of p-doping of the nanotubes. A schematic representation of the device is illustrated in Fig. 2; the catalytic medium shown in the figure consists of $[PdCl_2(PhCN)_2]$, $[^nBu_4N][NO_2]$ and $CuCl_2$ in benzyl alcohol; interestingly, removal of the $CuCl_2$ cocatalyst leads to enhanced device peformance.[12] The SWCNT matrix provides an electrical connection between two gold electrodes, and the sensor signal arises from a change in current between the electrodes under an applied voltage. The optimization of the conditions for ethene detection included functionalizing the SWCNTs with 4-pyridyl metal-binding sites; this proved to be a vital strategy as it ensures that the Pd centres are coordinatively bound to the nanotubes – presumably, the SWCNT-bound pyridine ligand replaces a PhCN ligand in $[PdCl_2(PhCN)_2]$. During testing, the sensor was exposed to different concentrations of ethene in samples of air, leading to a calculated limit of detection (LOD) of 15 ppb. After Swager and his coworkers had confirmed that the device was able to detect such low concentrations of ethene, the next step was to trial the device in an environment in which ethene was present as a plant hormone. Ethene is produced as flowers blossom, and the research team demonstrated the detection of ethene as carnations and lisianthus flowers opened. The Wacker/SWCNT-based sensor was able to reveal a peak in ethene production in just a few hours for carnations compared to a slower increase in ethene production over a few days for lisianthus flowers. As mentioned earlier, selective detection of ethene in the presence of other VOCs is essential for potential commercial applications of the sensor, and Swager demonstrated exceptional selectivity of the Wacker/SWCNT-based sensor for ethene versus a range of volatiles including acetone, acetonitrile, hexanes, simple alcohols, and toluene.

In this column, I have illustrated a didactic approach to introducing chemiresistive sensing of ethene into a discussion of the Pd-catalysed conversion of ethene to ethanal. This also provides an opportunity for presenting the structures and fundamental properies of carbon nanotubes.

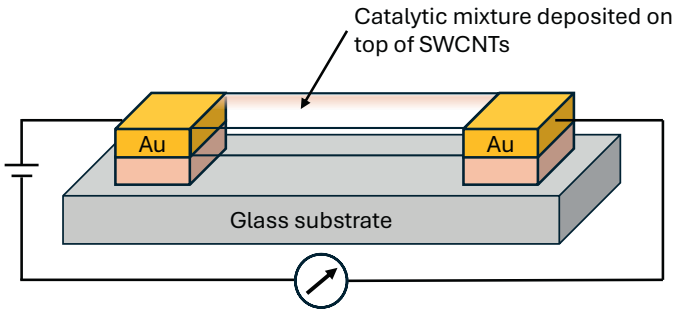

Fig. 2. Schematic representation of a chemiresistor with an insulating glass substrate, gold electrodes between which is a liquid catalytic mixture (see text) deposited on a SWCNT matrix. [The diagram is based on Fig. 1c in Ref. [12].]

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