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Reduction of aromatic and cyclic ketones to secondary alcohols – selected strategies and challenges

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Ketones constitute a fascinating class of organic compounds. Due to their structure, they show a significant similarity to aldehydes. They undergo reduction and oxidation reactions, as well as substitution reactions at the carbonyl group without further transformations and with the release of water. At the same time, considering steric hindrance (an aliphatic or aromatic substituent) or the presence of additional functional groups, the reactivity of ketones is generally lower than that of aldehydes. This paper presents methods for reducing selected aliphatic, aliphatic-aromatic and cyclic ketones only to corresponding secondary alcohols. These methods are essential due to the popularity of secondary alcohols in the broadly understood chemical and pharmaceutical industry. Reduction methods leading to other compounds, e.g. alkenes, are not presented in this publication.

Keywords: ketone, carbonyl group, reduction, efficiency, selectivity

INTRODUCTION

Ketones undergo chemical reactions similar to aldehydes (Fig. 1). These include nucleophilic addition reactions without further transformations. As a result of these reactions, cyanohydrins, sulfite compounds, and hydrates can be obtained [1]. The elimination of water follows the second group of reactions. This way, oximes, hydrazones, phenylhydrazones and semicarbazones can be obtained [2]. This group of reactions further includes those involving amines and can result in the formation of Schiff bases (in the case of primary amines) or enamines (in the case of secondary amines) [3]. In reactions between ketones and alcohols, hemi-acetals and acetals can be obtained [4]. These reactions proceed in two steps: first, an alcohol molecule is attached to the carbonyl group. Then, the resulting hemi-acetal reacts with a second alcohol molecule, forming the corresponding acetal by eliminating water.

Aldehydes and ketones also participate in reactions involving the addition of carbanions to the carbonyl group. As a result of these reactions, aldols and ketols can be formed [5]. It is also worth mentioning the addition of organomagnesium compounds to the carbonyl group, as a result of which primary, secondary, or tertiary alcohols can be obtained [6].

A significant difference in the reactivity of aldehydes and ketones can be observed in their oxidation reactions. Aldehydes are more susceptible to oxidation, and treatment with nitric acid, chromic acid, or potassium permanganate allows one to obtain the appropriate acid [7]. Selenium dioxide or selenium oxide may help oxidise unsaturated aldehydes. The spontaneous oxidation of aldehydes (auto-oxidation under the influence of air) is also worth mentioning. It is impossible not to mention the Cannizzaro reaction, a specific oxidation-reduction process for aldehydes without alpha protons. Ketones are much less susceptible to oxidation. Under the influence of strong oxidants, the bonds between the carbonyl atom

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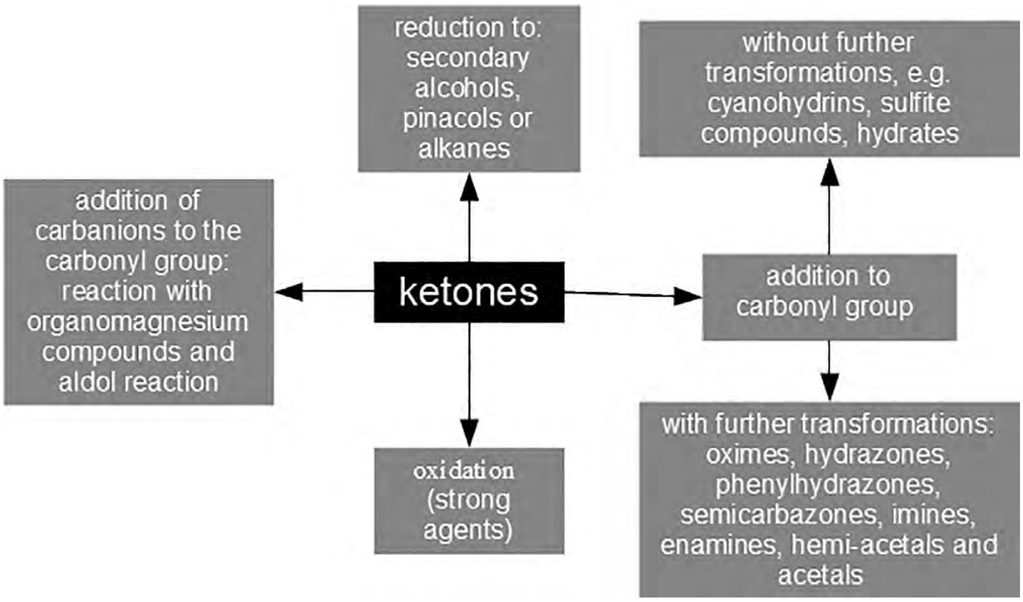


Fig. 1. Reactivity of ketones

and neighbouring atoms can be broken, forming a mixture of two carboxylic acids [8].

Popular agents for reducing the carbonyl group to a primary or secondary alcohol include sodium metal in alcohol, lithium aluminum hydride, sodium borohydride, and aluminum isopropoxide in isopropanol [9–10]. Specific reactions for ketones include the reaction leading to pinacols (e.g. with magnesium amalgam) or reactions leading to an alkane (e.g. the Wolff–Kiżner method using hydrazine or the Clemmensen method using zinc amalgam) [11–12].

The presented publication focuses on the possibility of reducing ketones to alcohols. The publication aimed to present reductions for selected ketones, rather than discussing the methods in a broader context. Ketone reduction methods are constantly evolving, and a publication analysing the methods would certainly be interesting. The topic of ketone reduction was presented more individually. An attempt was made to present various methods, exploring classical organic-chemistry reagents (e.g. Pd/ZrO_2 and $SmBr_2$), metal complexes (e.g. iridium and ruthenium), modern techniques (e.g. MAOS and sonification), or enzymes (carbonyl reductase).

The alcohols discussed in this publication (structures are presented in Fig. 2) are widely used in the chemical industry. They are used, among others, in:

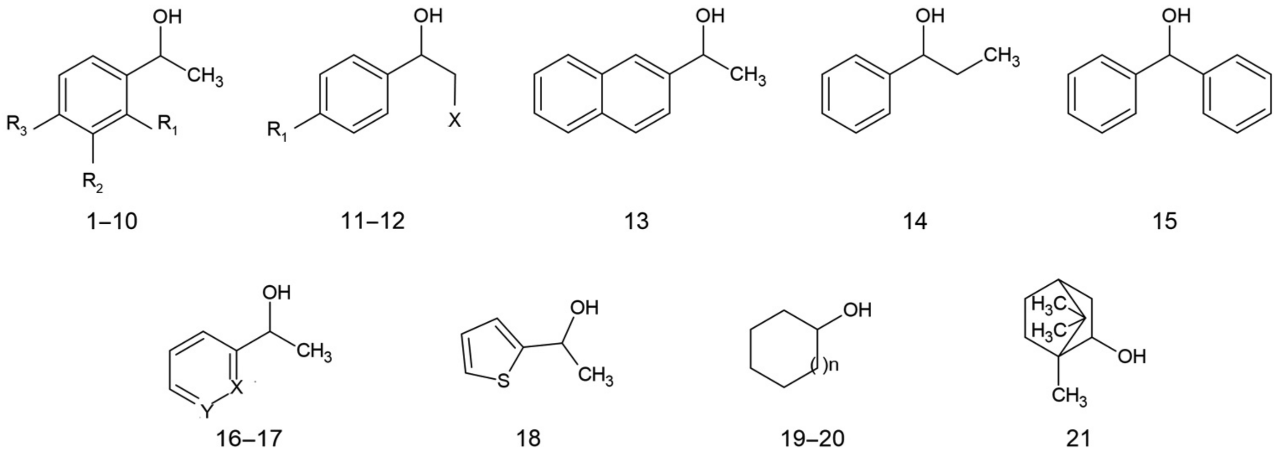


Fig. 2. Structures of the alcohols discussed in the publication obtained by reduction of ketones

a) the synthesis of drugs (e.g. rivastimgmine, doxylamine, and incadronate disodium) [13–15];

b) in industry as coating hardeners, paint plasticizers, and in the production of light-emitting diodes (OLEDs) and modern electrochromic materials [16–18];

c) for the production of fragrances [19–20];

d) in agriculture as fungicides, herbicides, growth regulators, and rooting stimulants [21–22].

1: $R_1 = R_2 = R_3 = H$
2–7: $R_1 = R_2 = H$, $R_3 = F, Cl, Br, NO_2, CH_3$ or OCH_3
8: $R_1 = NO_2$, $R_2 = R_3 = H$
9: $R_2 = H$, $R_1 = R_3 = CH_3$
10: $R_1 = H$, $R_2 = R_3 = OCH_3$
11: $R_1 = F$, $X = Br$
12: $R_1 = Cl$, $X = Br$
16: $X = H$, $Y = N$
17: $X = N$, $Y = H$
19: $n = 1$
20: $n = 2$

REDUCTION OF ACETOPHENONE DERIVATIVES

Methods of acetophenone reduction

Ketones can be reduced to secondary alcohols by metal catalysis. The method of exploring metal was based on using zinc as a reducing agent. Optimisation of the reaction (to determine the appropriate solvent, metal, proton source, time and temperature) was carried out using 4-CH_3-benzaldehyde. It was shown that the best results were achieved using Zn (5 eq.), 8 moles of NH_4Cl in 2 ml of THF (tetrahydrofuran), carrying out the reaction for 20 min at room temperature [23]. The tested aldehydes, regardless of a substituent nature or position, were reduced with good or excellent results. The ketones reacted more slowly, and the method's conditions had to be improved, among other changes, by increasing the temperature to 60°C. For acetophenone, the most advantageous use was 5 eq. Zn, 60°C, and the process time should be 3 h. By lowering the reaction temperature or shortening or extending the reaction time, worse results in 1-phenylethanol synthesis were obtained. The method also enables the high-efficiency (72–92%) reduction of 4-CH_3-acetophenone, 4-OCH_3-acetophenone, 4-Br-acetophenone, benzophenone, 2-acetylpyridine and cyclohexanone [23]. To sum up all the aspects characterising the described method, it can be stated that it allows, under mild conditions and in a fast way, the selective reduction of not only ketones but also aldehydes substituted with other functional groups, e.g. ester, nitrile, or epoxy, with an excellent reaction efficiency [23].

Another method for reducing acetophenone was a reaction with hydrogen using a bimetallic catalyst Pt–Pd/ZrO_2 carried out without a solvent. The model molecules for validating the method were cyclopentanone and acetophenone. The mass ratio of the individual metals constituting the catalyst and its type were studied to determine the effect on the reaction. The process conditions were set in advance, i.e. the time was 3 h, and the temperature was 160°C. An important aspect was reducing the pressure in the autoclave to 60 bar. It turned out to be interesting that acetophenone was reduced more effectively at 140°C than at 160°C. Secondly, while Pt/ZrO_2 proved to be a better catalyst for cyclopentanone, Pd/ZrO_2 was better for acetophenone. In addition, it was proven that the higher the palladium content in the Pt–Pd/ZrO_2 complex, the better the total reaction efficiency [24]. Another major issue is that 1-phenylethanol is not the only reaction product, as 1-cyclohexylethanol and cyclohexylmethylketone are also formed. At 160°C and 60 bar H_2, a study showed that the highest yield of 1-phenylethanol (64%) was obtained with Pd–Pt/ZrO_2 containing 25% palladium [24]. The lack of a solvent and the possibility of multiple uses of the catalyst make the method compatible with the direction in which modern chemical procedures are heading [24].

The third method is based on the use of many different ruthenium nanoparticles stabilised by aminoalcohols or pyrrolidines obtained from the transformation of (–)-(*S*)-verbenone [25], which is readily available in natural conditions. Based on the research, four types of stabilizers were selected, and their numbers were used to mark the chosen catalysts for the subsequent stages of the process, i.e. RuNPL15, RuNPL16, RuNPL18 and RuNPL21 (ruthenium nanoparticles). When acetophenone in heptane was reduced at 40 bar H_2 pressure regardless of the catalyst used, the yield of 1-phenylethanol was low despite the high overall process efficiency. It was mainly limited to obtaining by-products such as 1-cyclohexylethanol and 1-cyclohexylethan-1-one. Under the same reaction conditions, 4-OCH_3-acetophenone could be reduced using the RuNPL21 catalyst, yielding 1-(4-OCH_3-phenyl) ethanol with a yield of 21% [26]. It is intriguing that the hydrogenation of 4-NO_2–acetophenone leads only to the formation of amine derivatives, not alcohol. The procedure described briefly above in the context of obtaining secondary alcohols is not a method of high efficiency or selectivity. It mainly leads to hydrogenation of the aromatic ring [26].

Reduction by the reverse FLP (Frustrated Lewis Pairs) mechanism is worth mentioning. The idea for this innovative method of ketone reduction was initiated in 2014 when Ashley and Stephan developed the conventional FLP procedure. They used a strong Lewis acid (e.g. $[B(C_6F_5)_3]$) and a weak Brønsted base (e.g. dioxane or diethyl ether) to carry out the reaction [27]. Unfortunately, the method was highly selective for aliphatic ketones, while aromatic ketones underwent rapid side reactions. In 2018, a reverse version of this method was developed, using organic borate compounds as weak Lewis acids and $(pyrr)_3P = N\textit{t}Bu$ as a strong Brønsted base [28]. Optimisation led to the selection of eight different catalysts. Their effectiveness in reducing ketones was assessed. Another significant problem was selecting the appropriate molar ratio of acid to base, as increased acidification ensured higher activity and better repeatability of the process. The reduction of acetophenone and several of its derivatives (e.g. 4-OCH_3-acetophenone) was carried out, yielding 99% efficiency [28]. A noticeable aspect of the procedure is its long duration, which can extend to 40 h. Unfortunately, nitro derivatives of acetophenone did not react even at 100°C, probably because they deactivated the catalyst. Nevertheless, the reverse FLP method allows the reduction of not only aliphatic and aromatic ketones but also various functionalised N-, S-, or O-derivatives, such as 2-acetylthiophene and benzophenone, with yields over 90% [28].

Reduction of acetophenone, as the simplest aliphatic-aromatic ketone, can cause many problems for scientists. It is necessary to use the appropriate temperature and catalyst to increase the reaction's efficiency. Among the high-yield methods of reducing acetophenone to a secondary alcohol, it is also worth mentioning:

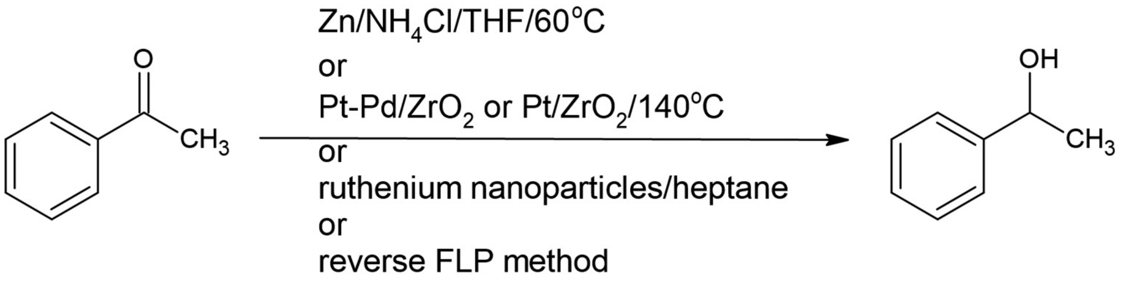
a) dimethylsulfide borane complex, N-[(2-Hydroxyphenyl)methyl]valine methyl ester in tetrahydrofuran at –77°C [29];

b) iridium compound $C_{25}H_{30}ClIrN_2O_3$, water, potassium hydroxide in isopropyl alcohol [30];

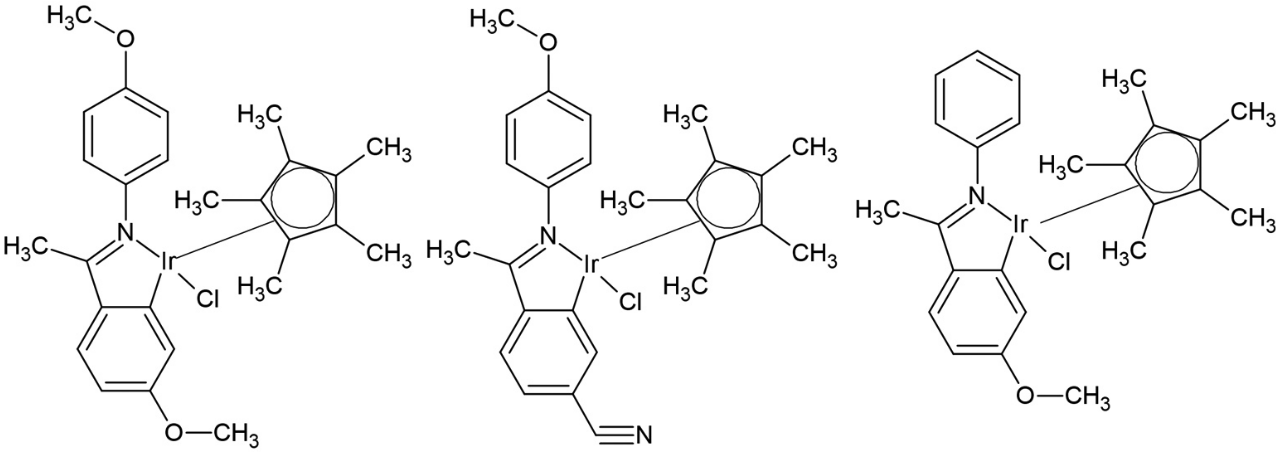
c) hydrogen in ethanol at 80°C; under 7500.75 Torr [31] (Scheme 1).

Reduction of haloderivatives of acetophenone

One of the major methods of hydrogenation of ketones is to use a wide range of metals or their complexes as catalysts. Currently, much attention is paid to ecological methods of reduction. In 2013, a study was published showing that *para*-substituted fluorine derivatives of acetophenone can be reduced efficiently to the corresponding secondary alcohols using organic iridium complexes. Initial experiments were carried out to hydrogenate amines using formic acid in methanol and metal catalyst No. 1 (presented in Fig. 3). Under those conditions, ketone reduction occurred, but very slowly. To optimise the reduction process, acetophenone was used, and the reaction was carried out in aqueous media with a mixture of HCOOH/HCOONa and two types of catalysts with different substituents on the aromatic ring. The key discovery was the effect of pH on the activity of



Scheme 1. Selected methods for the reduction of acetophenone to the corresponding secondary alcohol



Complex No. 1 Complex No. 2 Complex No. 3

Fig. 3. Structures of selected complexes (based on Ref. [32])

the reaction. Complex No. 1 showed the best reducing activity at pH 3.5, and complex No. 2 at a value oscillating around 2.5 (Fig. 3). Even small deviations in reaction acidity led to a significant decrease in efficiency. This phenomenon is probably due to the fact that iridium hydride, formed as a result of formate decarboxylation, can react in the low pH range with both the protons present in the reaction environment and with the selected ketone. The studies demonstrated an important relationship between the catalyst type and the need to maintain the appropriate acidity of the reaction mixture. Validated reactions on specific ketones were carried out using iridium complex No. 3, HCOOH/HCOONa mixture and H_2O, while maintaining a strongly acidic pH (ca. 2.5) and at 80°C [32] (Fig. 3).

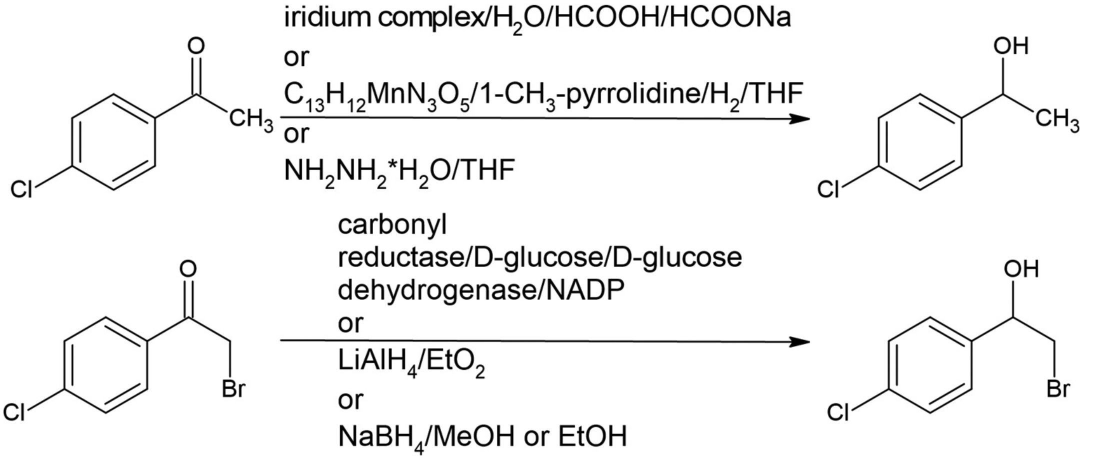
Table 1. **Comparison of the reduction efficiency of acetophenone and its *para*-fluoroderivatives based on the reaction time and substrate-to-catalyst ratio (S/C parameter) [32]**

Ketone	S/C	Reaction time, h	Efficacy, %
Acetophenone	2000	4	96
4-F-acetophenone	1000	12	89
4-Cl-acetophenone	500	12	89
4-Br-acetophenone	500	12	94

Analysing the results presented in the Table above, it can be concluded that halogen atoms in position 4 reduce the efficiency of the process, and the reaction time is extended threefold compared to acetophenone. The procedure using iridium complexes ensured a high efficiency of ketone reduction to alcohols [32].

Among the high-yield methods of reducing *p*-chloroacetophenone to a secondary alcohol, it is also worth mentioning: a) 1-methylpyrrolidine, compound $C_{13}H_{12}MnN_3O_5$ and hydrogen in tetrahydrofuran at 100°C and under 37503.8 Torr [33], b) complex $C_{36}H_{44}Cl_{Fe}N_4^{(1+)}*Cl_4Fe^{(1-)}$, potassium hydroxide in isopropyl alcohol at 82°C [34], c) sodium cyanoborohydride and microwave irradiation [35], d) hydrazine hydrate in tetrahydrofuran at 20°C [36]. Selected methodologies for the reduction of the discussed derivatives are presented in Scheme 2.

The chlorinated derivatives of acetophenone are also worth mentioning, which include 2-bromo-4'-fluoroacetophenone and 2-bromo-4'-chloroacetophenone. Obtaining halohydrins in the pharmaceutical industry is highly significant, as some are used as pure, optically active molecules in pharmacology [37]. Such medications are β-blockers used to treat cardiovascular diseases [38]. Carbonyl reductase isolated from *Candida magnoliae* – CMCR (*Candida magnoliae carbonyl reductase*) – was useful in obtaining alcohols from ketones. 2'-Br-4-F-acetophenone was selected for method validation, and the reaction was carried out using CMCR, D-glucose, D-glucose dehydrogenase, NADP (nicotinamide adenine dinucleotide phosphate), and phosphate buffer. The first studies were performed to select the appropriate solvent for the reaction, and it was shown that its absence significantly reduced the amount of alcohol obtained. The reason might be the ketone's poor solubility in the buffer. The effect of water-miscible and water-immiscible solvents on the reaction efficiency was then investigated.



Scheme 2. Selected methods for the reduction of *p*-chloroacetophenone and 2-bromo-4′-chloroacetophenone to the corresponding secondary alcohol

The best result was obtained using *tert*-butyl methyl ether. Studies on 2'-Br-4-Cl-acetophenone and 2'-Br-4-F-acetophenone showed that both compounds, when reduced under the same conditions, yield 82 and 89%, respectively [39]. The effect of the substituent on enzyme activity was interesting. It was proven that the presence of deactivating groups, i.e. electron-withdrawing groups (e.g. Cl and -F), increases the CMCR activity. The activating substituents, i.e. electron-donating groups (e.g. CH_3 group), reduce enzyme activity. The described biological method can be successfully used to synthesise (S)-α-bromohydrins without side reactions [39].

The frequently used reducing agents to obtain the discussed alcohols are $NaBH_4$ (e.g. in methanol and ethanol [40–41]) and $LiAlH_4$ (e.g. in diethyl ether [42]). Selected methodologies for the reduction of the discussed derivatives are presented in Scheme 2.

Reduction of 2-NO_2-acetophenone and 4-NO_2-acetophenone

Scientists are constantly looking for new methods of obtaining secondary alcohols from ketones. One of them is the use of iridium, iron, or ruthenium complexes [43–45]. Special attention should be given to recently developed aromatic compounds. Different ruthenium complexes were synthesised to produce alcohols. Complex A was used for validation studies on acetophenone (Fig. 4). Initial reactions using NaOH, KOH, or Na_2CO_3 showed that a strong base was necessary for TH (transfer hydrogenation). Considering the need to use potassium hydroxide, the isopropanol was selected as the best solvent. Then, under the already validated conditions, the appropriate catalyst was chosen to reduce acetophenone. It turned out that each of the developed complexes showed a reaction efficiency of over 80%. For both 2'-NO_2-acetophenone and 4-NO_2-acetophenone, the

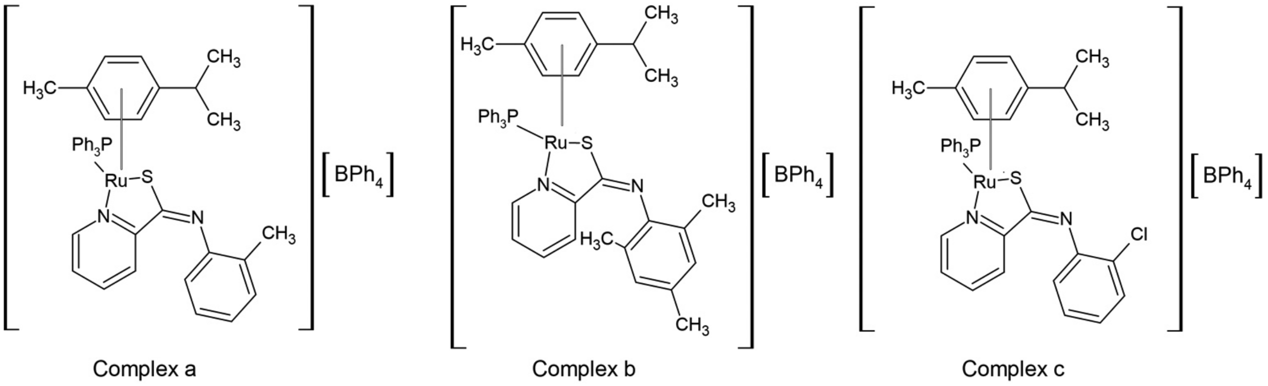


Fig. 4. Structures of selected complexes (based on Ref. [46])

reaction efficiency was 100% [46]. Interestingly, other ketones, e.g. 2-acetylpyridine, 2-acetylthiophene and 4-Br-acetophenone, were also reduced (efficiency above 90%) [46]. The presented results mean that the described method can be assessed positively not only due to its high efficiency in ketone hydrogenation, but also from the economic and ecological point of view, because the developed ruthenium complexes can be successfully used even three times without a significant loss of properties [46].

The reactivity of the nitro group forces scientists to use selective reducing agents. In addition to the above-mentioned complex compounds, the following can be used for the selective reduction of *p*-nitroacetophenone:

a) formic acid, triethylamine in water [47],

b) potassium *t*-butylate in isopropyl alcohol [48],

c) ammonia borane in tetrahydrofuran [49],

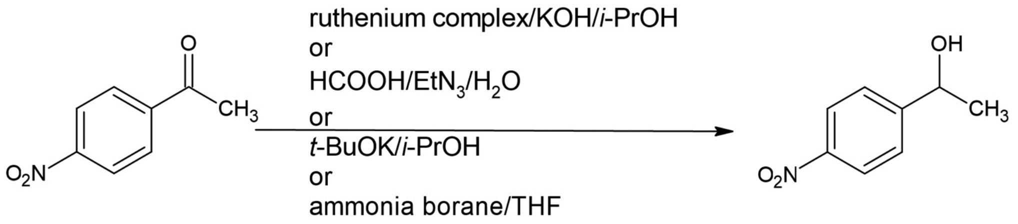
d) zirconocene dichloride, (dimethoxy)methylsilane and diethylamine in toluene [50].

Scheme 3 shows the selected methods for the reduction of *p*-nitroacetophenone.

Reduction of 4-CH_3-acetophenone and 2,4-diCH_3-acetophenone

With the development of a new direction in chemical procedures, the 'Green Chemistry', a search began for not only effective but also safe methods of synthesising secondary alcohols [51]. The concept of using HHP (high hydrostatic pressure) was developed based on two discoveries, i.e. the Begius and Haber–Bosch processes, for which the two scientists were awarded the Nobel Prize in 1931 [52]. One element of an ecological approach to the synthesis of chemical compounds is, among others, the elimination of harmful organic solvents and the use of water instead [53]. Initially, research on reducing carbonyl compounds in aqueous environments was conducted using aluminum alloys with other metals. The original procedures were based on the hydrogenation of acetophenone using a Ni–Al alloy in water by using microwave radiation. However, this process did not lead to selective synthesis of alcohols, because at elevated temperatures the metal alloy became too reactive [54]. To develop an effective method for reducing ketones, each of the two substrates was subjected to four reactions differing in time, temperature, pressure, and radiation source, while the common element was the use of a nickel–aluminum alloy as a catalyst [55].

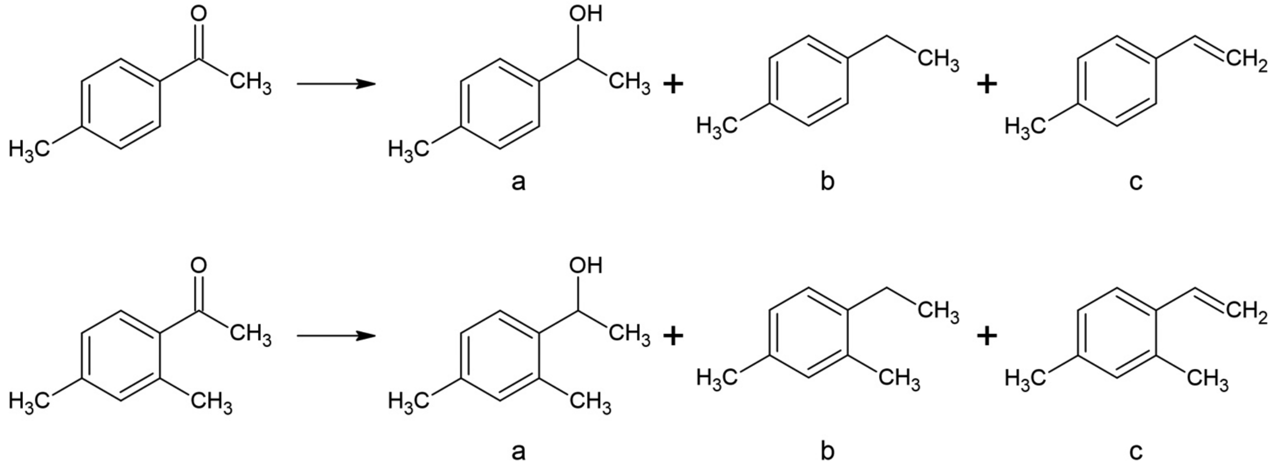
Table 2 and Scheme 4 show that 4-CH_3-acetophenone is reduced selectively to the corresponding alcohol under normal pressure. In turn, 2,4-diCH_3-acetophenone is better reduced under lowered hydrostatic pressure. Interestingly, sonification has proven to be the most selective method



Scheme 3. Selected methods for the reduction of *p*-nitroacetophenone to the corresponding secondary alcohol

Table 2. **Comparison of conversion and selectivity results for obtaining alcohols from methyl derivatives of acetophenone depending on the energy source used, pressure, time and reaction temperature [55]**

Ketone	Energy	Pressure, kbar	Time, h	Temperature, °C	Conversion, %	Selectivity, %
4-CH_3-acetophenone	–	–	8	40	100	80
	–	2.8	8	40	100	95
	Sonification	–	2	25	47	100
	Microwave irradiation	–	2	80	100	0
2,4-diCH_3-acetophenone	–	–	8	40	100	80
	–	–	7	40	97	97
	–	2.8	7	40	100	94
	Sonification	–	2	25	48	100
	Microwave irradiation	–	2	80	0	0



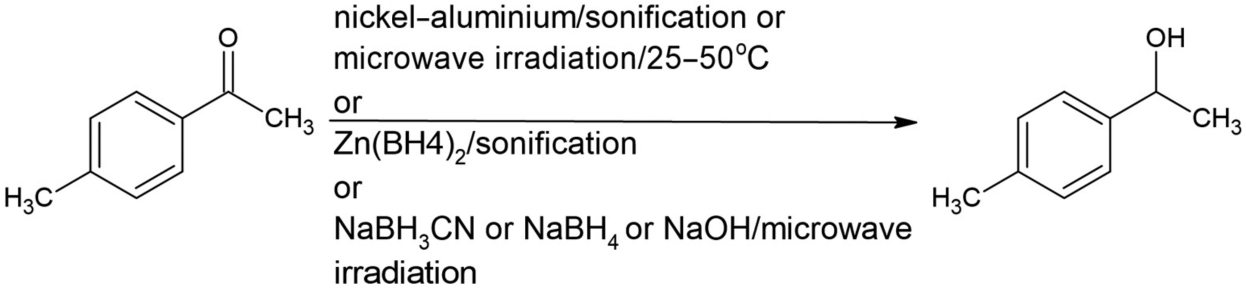
Scheme 4. Possible reduction products of 4-methylacetophenone and 2,4-dimethylacetophenone. Acoording to Ref. [55]

of obtaining alcohols, but the presented efficiency is relatively low. Microwave procedures were ineffective at reducing the discussed ketones. To sum up, the described methodology using HHT allows for obtaining good TH ketone results and is additionally compatible with the assumptions of 'Green Chemistry' [55].

The pro-ecological approach to *p*-methylacetophenone reduction has also been used in microwave reactions (with sodium cyanoborohydride [25], sodium borohydride [56], or sodium hydroxide [57]) or by sonication [58]. The methodologies discussed are presented in Scheme 5.

Reduction of 4-OCH$_3$-acetophenone and 3,4-diOCH$_3$-acetophenone

In 2022, a method was developed that paradoxically does not require a catalyst for the reaction. The essence of the process is UV light irradiation in an alkaline environment. This development corresponded to an ecological approach to chemical synthesis. Initial studies were conducted on olefins, specifically 1,1-diphenylethylene, using a lamp generating UV–C radiation with a power of 32 W and a wavelength of 254 nm. When selecting a solvent for the reaction, it turned out that only DMF (dimethylformamide) allows one to obtain alcohols. Then, based on the knowledge that adding an inorganic base can improve electron transfer, various amines and pyrrolidine were tested alone or in combination with potassium hydroxide. Alkalisation of the reaction mixture significantly improved the reaction rate. After validation of the methodology on olefins, attention was focused on ketones, specifically benzophenone and acetophenone. An interesting fact was that the latter of the mentioned compounds is better reduced in the presence of UV–C than UV–A, due to stronger UV–C absorption. The conditions had to be slightly modified to hydrogenate methoxy derivatives of acetophenone. For example, the reaction time was shortened to 12 h, and DMF was used as the solvent, with pyrrolidine and potassium hydroxide as the alkalising agents. 1-(4-OCH$_3$-phenyl)ethan-1-ol and 1-(3,4-diOCH$_3$-phenyl)ethan-1-ol were obtained in



Scheme 5. Selected methods for the reduction of *p*-methylacetophenone to the corresponding secondary alcohol

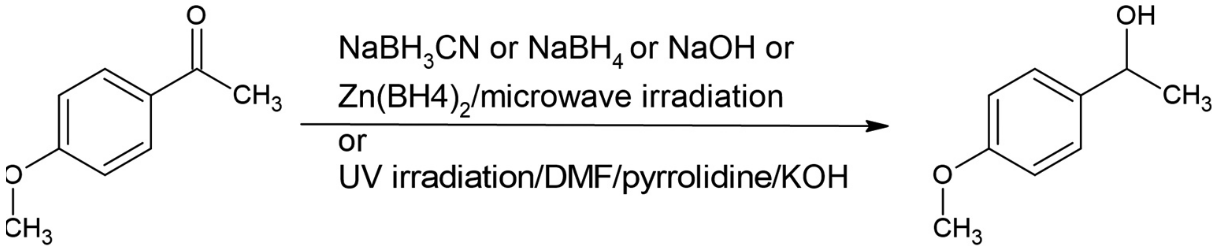
yields of 65 and 67%, respectively [59]. Interestingly, compounds containing electron-repelling groups in the ring in the *para* position, e.g. F (74%), were reduced slightly better, while those donating electrons, e.g. CH_3 (55%), were reduced worse. What is particularly noteworthy about the presented methodology is that it does not require the use of a classical reducing agent and catalyst and enables not only the hydrogenation of compounds but also their deuteration [59].

It is worth emphasising that microwave techniques (especially with the addition of sodium cyanoborohydride [35], sodium tetrahydroborate [60], zinc (II) tetrahydroborate [61], or sodium hydroxide [57]), are also widely used for the reduction of *p*-methoxyacetophenone. Selected methods for reducing this ketone are shown in Scheme 6.

METHODS OF REDUCING 2-ACETYLNAPHTHALENE

The first described reaction involves iridium complexes in the presence of ammonium formate. The Leuckart–Wallach (LW) reaction involves the formation of primary amines from aldehydes using $HCOONH_4$ at elevated temperatures, without a catalyst [62]. This method is not widely used today due to the poor chemoselectivity of the products, including a secondary alcohol. Based on the salt mentioned above, strategies to improve reaction parameters were initiated, which could be used to synthesise amines or alcohols. For this purpose, iridium complexes of different cyclic structures were created. The first of them, called Ir-L1 and Ir-L2, were synthesised by treating N,N'-dimethylurea with chlorophosphine or phosphorochloridatein (Fig. 5) [63]. Other metal structures, called cyclometallic iridium complexes, have a relatively similar structure to the one mentioned above, with the difference that they do not contain phosphorus connected to the central atom [64]. Control tests were performed on 2-acetylnaphthalene in both cases, and the best reaction conditions, catalyst and solvent were selected [63–64].

The best reaction environment in method 1 was provided by HFIP (hexafluoroisopropanol), and catalyst 1 was considered more efficient [63]. In the second case, a mixture of water and azeotrope of formic acid with triethylamine (F/T) and complex 1i [64] was applied. The common point between the two methods is the use of $HCOONH_4$, while the time, temperature and process yields differ significantly.



Scheme 6. Selected methods for the reduction of *p*-methoxyacetophenone to the corresponding secondary alcohol

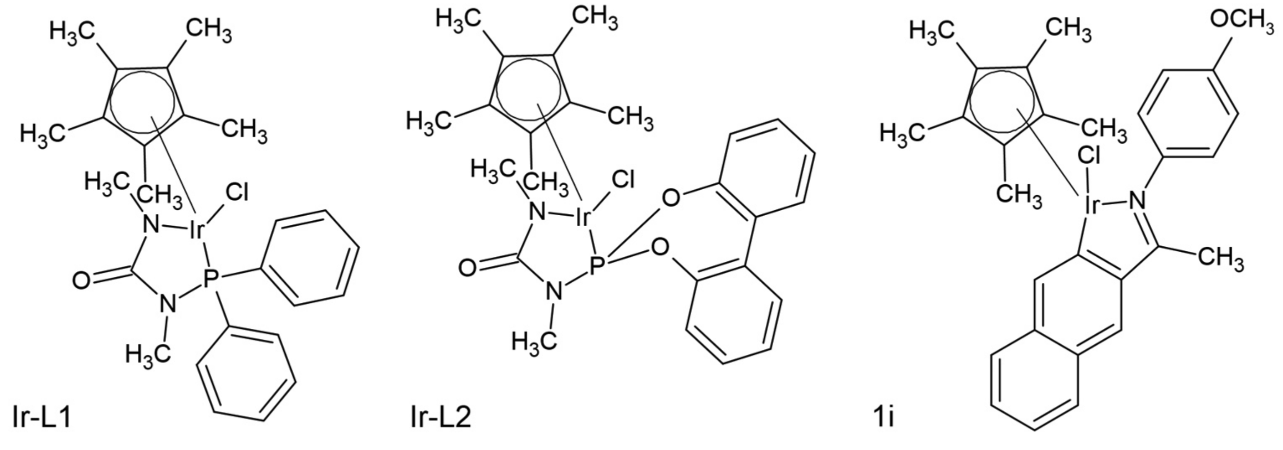


Fig. 5. Iridium complexes applied in alcohol synthesis (based on Refs. [63] and [64])

Table 3. Presentation of the most favourable reaction conditions for the reduction of 2-acetylnaphthalene using method number 1 [63]

Method 1				
Catalyst	Co-reagents	Solvent	Time, temperature	Reduction to alcohol, %
cat. Ir-L1	ammonium formate	HFIP	18 h, 50°C	31

Table 4. Presentation of the most favourable reaction conditions for the reduction of 2-acetylnaphthalene using method number 2 [63]

Method 2				
Catalyst	Co-reagents	Solvent	Time, temperature	Reduction to alcohol, %
cat. 1i	ammonium formate; F/T; HCO_2H/Et_3N (5:2)	water	4 h, 80°C	98

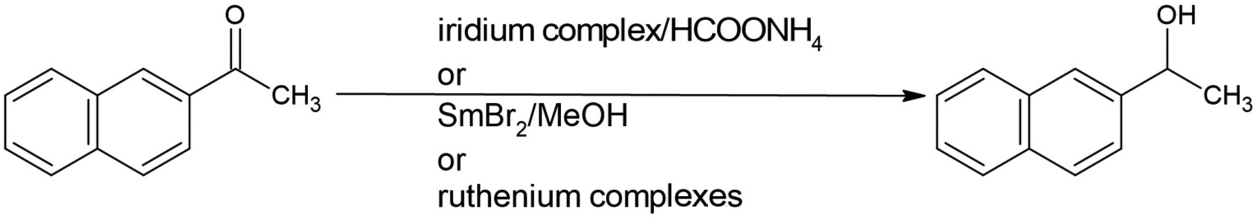
The first method is characterised by a long reaction time (18 h) and a low yield of secondary alcohol (31%) [63]. In turn, the second procedure requires a slightly higher temperature, i.e. 80°C, but the reduction time is shortened to 4 h, and the yield of 1-(2-naphthyl)ethanol synthesis reaches 98% [64]. In summary, from the perspective of obtaining the described alcohol, method 2 is more advantageous because the presented results are higher and the process time is significantly shorter. In turn, method 1 can be considered more effective for the synthesis of primary amines [63–64].

The method uses samarium and allyl bromide in methanol and requires maintaining room temperature. Its products can be both alcohols and pinacols. Based on precursory studies using acetophenone, it has been shown that the key to obtaining a given result with an exemplary efficiency is the appropriate ratio of both substrates catalysing the process. Preliminary preparation of samarium dibromide was also considered instead of using a binary reducing mixture, but it is sensitive to air, and its synthesis is relatively complicated. The hydrogenation efficiency of 2-acetylnaphthalene is low, ca. 24% [65]. Despite this, it is the best result among the other ketones tested, because, for example, 4-CH_3-acetophenone and 4-Cl-acetophenone were reduced to 11 and 8%, respectively. The main focus of the described procedure is pinacolisation reactions that are more efficient than competitive alcohol synthesis [65].

High-yielding complexes that allow obtaining the discussed alcohol also include ruthenium compounds [66–68]. Naphthalene can be reduced to a secondary alcohol using the methods summarised in Scheme 7.

METHODS OF PROPIOPHENONE REDUCTION

Studies were carried out using sodium borohydride. They aimed to determine how aluminum chloride would affect the reaction's quality and the quantitative result. For this purpose, acetophenone was used as a reference substrate, which, as it turned out, was reduced to the desired alcohol by only 5% [69]. The low result is probably due to the formation of 1-phenylethanol from the reaction of borohydride with the ketone, which subsequently forms a complex with the chloride used. The final and main product of the process is ultimately ethylbenzene. Unfortunately, the situation is similar to propiophenone, where the synthesis of propylbenzene is observed mainly, not 1-phenyl-propanol (7%) [69]. The results were also compared with those obtained with the combination of lithium tetrahydroaluminate and aluminum chloride. It was shown that the reduction with $LiAlH_4$ was inefficient and required a nitrogen flow. The above methodology would not be suitable for performing the reduction processes



Scheme 7. Selected methods for the reduction of 2-acetylnaphthalene to the corresponding secondary alcohol

essential to this work, as the main goal is to obtain 1-phenylpropanol, which is formed in low amounts [69]. An innovative method currently being developed to catalyse reduction is the use of metal nanoparticles incorporated into porous structures. It consists, first, of infiltrating or sublimating the metal precursor into the porous network. Then, the metal is brought into nanoparticles, e.g. by using hydrogen [70]. The procedure for synthesising such a catalyst is called MOCVD (metal-organic chemical vapour deposition). Various substrates were developed at the initial research stage, where palladium was deposited, and subsequent process phases were performed. LL4842 and HH1545 were selected to reduce selected ketones, and the results are presented in the Table below [71].

Based on these results, it can be concluded that HH1545 is a better catalyst for obtaining 1-phenylethanol and 1-phenyl-propanol. The reaction efficiency (reaching up to 100%) does not correspond to selectivity in the context of alcohol synthesis [71]. It was also noted that deoxygenation occurs more quickly than reduction. It is interesting that, for example, using LL4842 results in higher total hydrogenation results for propiophenone than for 2,2-dimethylpropiophenone. The more space the substrate occupies, the more difficult it is for palladium nanoparticles to access it, which translates into a lower utilisation of the catalytic potential. Under appropriate conditions, the above methodology will provide excellent results in obtaining alcohol. However, it is worth emphasising that, despite the possibility of recycling catalysts, their decomposition time is very long and favours a prolonged reduction, which may not necessarily improve reaction results [71].

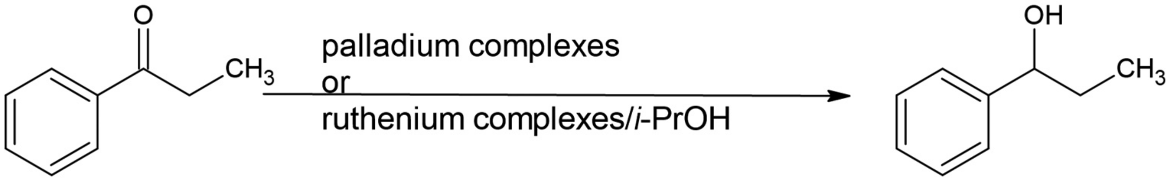
Moreover, the selective reduction of propiophenone to the corresponding alcohol can be carried out, among others, by ruthenium complexes in isopropanol [72–73]. Scheme 8 presents the methodologies for obtaining a secondary alcohol from propiophenone.

BENZOPHENONE REDUCTION METHODS

Ketones, as popular organic compounds, not only undergo reduction reactions but also, for example, deoxyhalogenation. The problem occurs when the above processes occur in parallel, depending on the reagent proportions. Most halides produced in the industry are chlorides, and their synthesis involves the use of corrosive and harmful substances, e.g. thionyl chloride or triphosgene [74]. $TiCl_4$ as a Lewis acid was proposed to eliminate the above reagents. $TiCl_4$ acts as a carbonyl activator and halogen carrier, and borazine acts as a reducing agent [75–76]. The main goal of the reaction was to obtain the final product as an organic chloride while maintaining appropriate conditions; it could also lead to the hydrogenation of the ketone and, thus, to the formation of a secondary alcohol. In control tests, benzophenone and isobutyrophenone were used as substrates. In both cases, it was shown that the lower the concentrations of the halogen carrier and reducing agent in the reaction, the more alcohol was obtained. Increasing the concentration of the reagents leads to less alcohol and more chloride and aromatic compounds. Interestingly, isobutyrophenone undergoes hydrogenation more readily, even at higher reagent concentrations, and the reaction time is shorter than for benzophenone.

Table 5. Table comparing the efficiency of catalysts LL4842 and HH1545 for the reduction of acetophenone and propiophenone [71]

Ketone	LL4842- catalyst number 1		HH1545- catalyst number 2		
	Conversion to alcohol (%) at 80°C	Conversion to alcohol (%) at 50°C	Conversion to alcohol (%) at 80°C	Conversion to alcohol (%) at 50°C	Conversion to alcohol (%) at 39°C
Acetophenone	<1	32	<1	90	98
Propiophenone	<1	80	10	92	97



Scheme 8. Selected methods for the reduction of propiophenone to the corresponding secondary alcohol

Benzophenone was reduced after 40 h, yielding 42% [76]. The change in the concentration equilibrium favours the formation of 1-Cl-2-CH_3-phenylpropane in most cases, and the reduction process competes with the chlorination reaction. In summary, the described method, due to the long hydrogenation time of benzophenone and the need to control the concentration of reagents, does not allow for obtaining high alcohol yields [76].

Depositing inorganic metal nanoparticles, e.g. palladium, onto an organic structure could increase the selectivity of transfer hydrogenation [77–80]. To improve the reduction, a procedure was developed that involved placing highly catalytically active metals in the micropores of zeolites. Two methods were designed to reduce benzophenone. The first used the Pd@Beta, i.e. palladium deposited on a specific zeolite type. The second method used a combination of metal and carbon. An essential element of both reactions was the use of elevated temperature and 1 MPa H_2 pressure [78].

The tabulated results show that the desired reaction product, i.e. diphenylmethanol, is formed in both cases with a relatively low efficiency. The publication's authors argue that the Pd/C catalyst is better suited for reducing benzophenone to a secondary alcohol because the C=O group is easily accessible. In turn, Pd@Beta has micropores that control steric adsorption, so the benzene ring is more readily hydrogenated than the ketone group. The description of the above reaction showed that obtaining diphenylmethanol by reducing benzophenone does not require complex modifications of palladium nanoparticles, in fact, placing them on activated carbon is more efficacious‘ [78].

Palladium catalyst supported on carbon is often used in the reduction of benzophenone [79–80]. Selected methods of benzophenone reduction are summarised in Scheme 9.

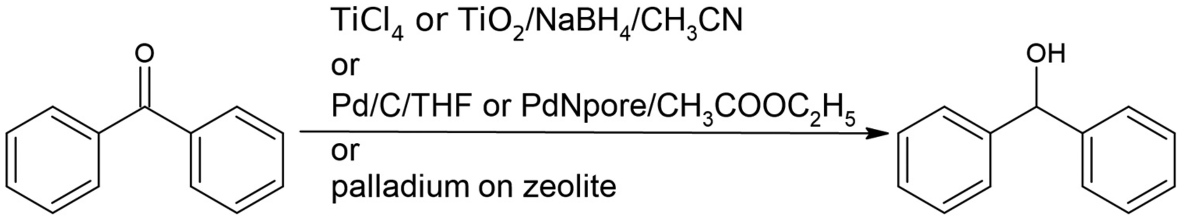
REDUCTION OF HETEROAROMATIC ACETYL DERIVATIVES

Methods of reducing 2-acetylpyridine and 3-acetylpyridine

Meerwein–Poondorf–Verley transfer hydrogenation (MVP) is a model reaction of reducing ketones to secondary alcohols using a metal alkoxide, specifically aluminum isopropoxide, and 2-propanol [81]. However, as science developed, it was found that this process could also be carried out using salts of certain metals, for example, those of the lanthanide group [82]. In parallel, research was conducted using metal-catalytic systems, e.g. iron in an alkaline environment, with *i*-PrOH as a proton source [83]. Interestingly, during the first reactions, it turned out that the reduction of a ketone to an alcohol is possible without a catalyst. The presence of an alkali alkoxide is all that is necessary. Therefore, reactions were performed on acetophenone using, among others, *i*-PrONa, *i*-PrOLi, or potassium *t*-butoxide, with propane-2-ol as a proton donor. The results showed that, at 120°C, sodium or lithium isopropoxide reduced the ketone with 71% efficiency within one and a half hours, whereas potassium *t*-butoxide under the same conditions decreased the ketone to 20% [84]. Next, the influence of temperature on the process was evaluated, and the reaction mixture was heated to 180°C under microwave irradiation. The study showed that

Table 6. **Comparison of qualitative and quantitative results of reactions carried out using Pd@Beta and Pd/C catalysts [78]**

Catalyst	Conversion, %	Reduction products, %
Pd@Beta	62.0	Cyclohexyl(phenyl)methanone [92.0] and diphenylmethanol [7.8]
Pd/C	89.1	Diphenylmethane [48.6] and diphenylmethanol [43.4]



Scheme 9. Selected methods for the reduction of benzophenone to the corresponding secondary alcohol

reaction time was reduced to 20 min and efficiency increased to 96% [84]. Interestingly, the MPV method proved ineffective for the hydrogenation of pyridine derivatives to the corresponding alcohols. However, the described procedure under validated conditions, i.e. with the addition of catalytic amounts of *i*-PrOLi, showed 99% efficiency [84]. Additionally, exciting studies were carried out on bromine-substituted acetophenone derivatives. The presence of a halogen in the *para* position has been shown to weaken the reaction compared to the *ortho* or *meta* isomers. This is explained by the stronger electronic effect resulting from the presence of a deactivating substituent (e.g. Br) in the *para* position than the steric effect resulting from *ortho* or *meta* substitution.

It is worth emphasising the use of isopropanol for the reduction of 3-acetylpyridine, because it is combined with molecules such as:

a) ammonia borane and $CoCl_2$(*t*-Bu-DAB) [85],

b) $C_{47}H_{44}N_6O_2P_2Ru^{(2+)*}2F_6P^{(1-)}$ and potassium hydroxide [86],

c) $C_{19}H_{33}ClIrN_3O$ and sodium isopropylate [87],

d) potassium *t*-butylate [88],

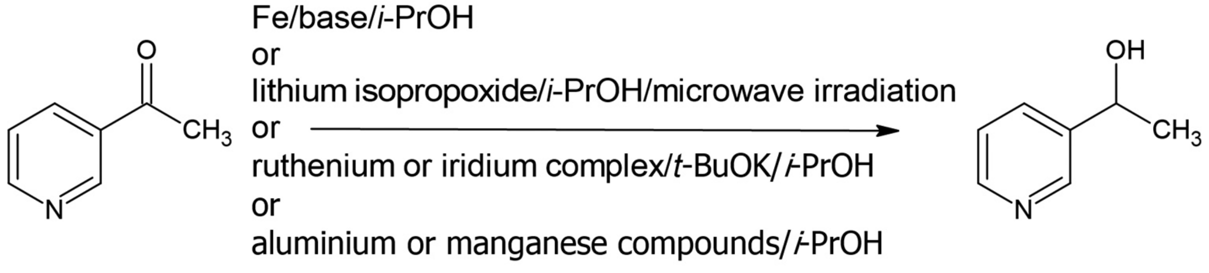
e) $C_{36}H_{103}AlO_4Si_{14}$ [89].

Methods for the reduction of 3-acetylpyridine are shown in Scheme 10.

Methods of 2-acetylthiophene reduction

In 2004, an article reported the reduction of 2-acetylthiophene using metal iodides, specifically SmI_2 or TmI_2, with or without HMPA (hexamethylphosphoramide). Hexamethylphosphoramide is a proven agent that increases the reduction potential of samarium iodide, but it is suspected to induce cancer development [90–91]. To determine the best method of synthesis of 1-(thiophen-2-yl) ethanol, three different reactions were carried out using samarium iodide in THF (method A), samarium iodide in THF with the addition of HMPA (method B), and thulium iodide in THF (method C). The first stage of the reaction is the formation of an intermediate nucleophilic compound *via* electron transfer from the metal salt. Then, a proton is abstracted from tetrahydrofuran. Based on the studies, procedure A reduces the discussed ketone with an efficiency of 87%. The remaining methods, i.e. B and C, yield worse results, below 50% [92]. The publication also discussed methods of cross-coupling between different aromatic hydrocarbons, leading to obtaining, among others, pinacols. The above fact is significant in the context of catalyst selection because, while samarium iodide favours ketone reduction, thulium iodide performs better in the coupling reaction. Depending on the desired outcome, the described method offers an excellent potential for producing a broad spectrum of products using 2-acetylthiophene as the substrate [92]. The subject of transfer hydrogenation has already been discussed in this paper in the context of an efficient and effective method of ketone reduction. It involves using a proton donor (other than hydrogen) and a metal as a catalyst [93]. Research has begun on organometallic catalysts based on Rh, Ru, or Ir to develop more environmentally friendly reagents [94].

Ruthenium complexes have not developed much in terms of applications in chemical synthesis. Hence, the publication attempted to learn about and improve their properties for potential use in reduction processes. It turned out that homogeneous analogues are less active than those with a wide range of ligands [95]. Of particular interest to researchers were carbon nanotubes based on graphene, from which SWNTs (single-walled carbon nanotubes) were created; on them, a ruthenium dimer was deposited, creating a highly effective catalyst [96]. This combination's essence was the metal complex's linker with



Scheme 10. Selected methods for the reduction of 3-acetylpyridine to the corresponding secondary alcohol

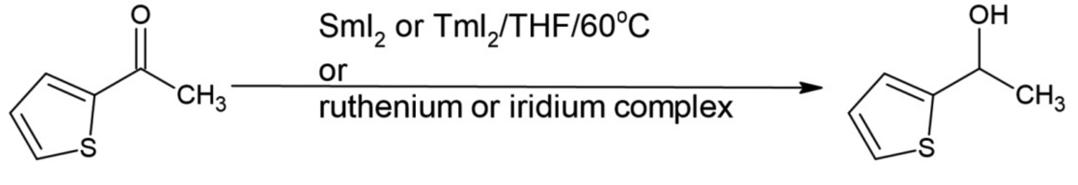
the organic one, i.e. aniline. The acetophenone was used to confirm the reduction properties of the above catalyst. *i*-PrOH was used as a proton source and solvent, and the reaction mixture was alkalised with a potassium base. It turned out that the most effective catalysing agent for TH will be SWNT-NH_2-Ru because the process efficiency is 95% [96]. In comparison, despite the increased reaction time, the absence of metal in the complex results in a significant decrease in reduction efficiency [96]. It is also worth noting that other ruthenium complexes also gave high TH efficiency results, but the TON (turnover number) and TOF (time of flight) results were lower, and the reaction time was much longer. Under validated process conditions, 2-acetylthiophene was also hydrogenated, and 1-(thiophene-2-yl)ethanol was obtained with a result of 98% [97]. The methodology also allows the preparation of many other secondary alcohols described in this theoretical part, i.e. 1-phenylethanol (93%) or 1-(4-Br-phenyl)-ethan-1-ol (>99%) [97]. Interestingly, there are better processes than this, as 4-OCH_3-acetophenone and benzophenone are reduced in this way with a poor efficiency, yielding results below 25% [97]. The described metal complex has several advantages, i.e. the possibility of multiple use, high specificity, selectivity and stability. However, the disadvantage of the process is the long reaction time, usually 16 h [97].

Scheme 11 shows the possibilities of reducing 2-acetylthiophene to the corresponding secondary alcohol.

METHODS FOR REDUCING CYCLOHEXANONE AND CYCLOHEPTANONE

Firstly, a method with the iron (II) Schiff base deposited on mont-K10 (Fe@imine-mont-K10) should be described. The ecological approach in the context of broadly understood chemistry has become a common trend. Limiting the use of toxic noble metals as catalysts has also become a primary task for synthetic chemists. The key here is the possibility of using environmentally friendly elements, such as Fe [98]. Based on this knowledge, an iron (II) salt was deposited on montmorillonite K10 clay. The substrate used has several advantages, including a low toxicity, non-corrosivenes and and recyclability [99]. The benzophenone was used as the starting substrate to select suitable reaction conditions. Using the proper solvent, an alkalising agent, temperature, process time, and catalyst concentration was crucial. Sodium isopropoxide was chosen as the base, and the environment was a mixture of acetonitrile and *i*-PrOH. Interestingly, preliminary studies showed that the catalyst is inactive at room temperature, and the reaction must be conducted at 80°C. The optimal process time was 5 h. Cyclohexanone and cycloheptanone were reduced with an efficiency of 99% [100]. The difference between them, however, was the hydrogenation time: 1 h for the first ketone and 30 min for the second. The publication also reports the possibility of synthesising secondary alcohols from acetophenone and its derivatives, but the reaction time exceeds 3 h. Analysing the above methodology, it can be concluded that it is characterised by high reduction efficiency. Still, it should be noted that, according to the authors of the publication, the described catalyst can be used in synthesis up to five times without losing its activity [100]. A significant advantage of procedures using lamps that generate light of appropriate power is their environmental safety. The described method of reducing ketones to secondary alcohols uses radiation generated by a high-pressure mercury lamp and a proton donor solvent [101].

Based on the results presented in Table 7, it can be concluded that only the method exploring isopropanol is suitable for synthesising cyclohexanol. Other reduction attempts led to the formation of 1,2-diols and acetals. In turn, cycloheptanone was reduced to cycloheptanol using methanol, while this process yielded only 15% [86]. The hydrogenation results are low, and the reaction time,



Scheme 11. Selected methods for the reduction of 2-acetylthiophene to the corresponding secondary alcohol

Table 7. **Comparison of cyclohexanone and cycloheptanone reduction efficiency depending on the solvent used, reaction time and high-pressure mercury lamp power [101]**

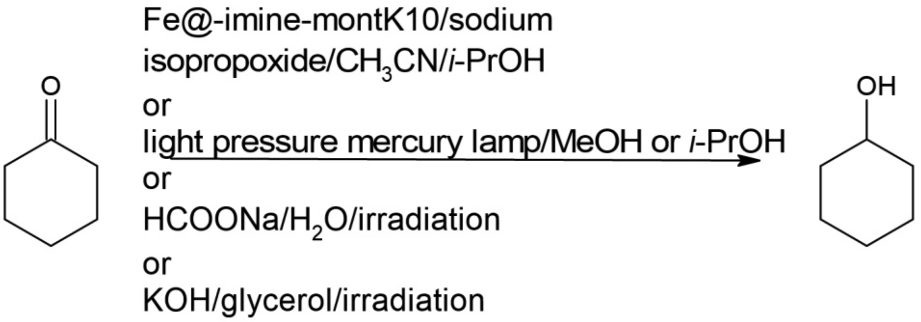
Ketone	Solvent	Mercury lamp power, W	Time, h	Alcohol yield, %
Cyclohexanone	MeOH	400	4	–
	MeOH	100	18	–
	MeOH:water (20:1)	100	22	–
	i-PrOH	100	20	62
Cycloheptanone	MeOH	400	4	15

e.g. for cyclohexanone, is very long; hence, it is not a standard method for the synthesis of alcohols, but it can be successfully used in acetylation or cross-coupling reactions [101].

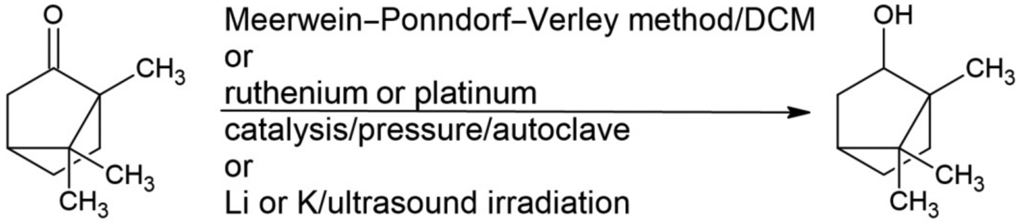
It is worth emphasising that this is not the only technique using radiation to obtain cyclohexanol [102–103]. Example methodologies for obtaining cyclohexanol are presented in Scheme 12.

CAMPHOR REDUCTION METHODS

A publication from 1997 by Cha and Kwon [104] provided at that time extremely significant information concerning the transfer hydrogenation of ketones using Al-isopropoxydiisobutylate in diethyl ether. The reaction was supposed to proceed on a principle similar to the well-known Meerwein–Ponndorf–Verley reduction. The evidence was the fact that the level of thermodynamically stable alcohol increased during the process [104]. However, the disadvantage of this method was its long duration, which could take up to several days. In their efforts to improve this process, scientists began research using the same catalyst while analysing the effects of the solvent on yield, selectivity, and reaction time. The aim of validating the procedure was to obtain the highest possible level of equatorial alcohol compared to axial alcohol, because the former is much more thermodynamically stable. Based on this knowledge, hydrogenation of camphor was carried out in tetrahydrofuran, dichloromethane and toluene. In THF, the ketone could not be reduced, while the test in DCM (dichloromethane) achieved a result of 83%, of which the ratio of equatorial and axial alcohols was more stable [105]. In turn, the results in toluene were lower than in DCM and favoured the synthesis of less favourable alcohol. To sum up, the change of solvent significantly influenced not only the efficiency of alcohol synthesis but also reduced the reaction time, which was shortened to 6 h [105]. Another procedure is based on the hydrogenation of the ketone in an autoclave under pressure of 8 MPa H_2, at a temperature of 50°C, using ruthenium or platinum catalysts. The reactions were conducted primarily to assess the effect of metal dispersion on a 5% carbon support on the reduction efficiency. The results were compared to those obtained using only metal catalysts. It was shown that platinum compounds will not reduce camphor to its alcohol equivalent, regardless of the form of the metal used. On the other hand, studies using ruthenium showed that it catalyses reactions more effectively when supported on a 5% carbon support, with a total process efficiency of 94% [106]. The main alcohol synthesised was the *exo* form (37%). For comparison, reducing acetophenone to 1-phenylethanol, regardless of the metal used or its dispersion on the support,



Scheme 12. Selected methods for the reduction of cyclohexanone to the corresponding secondary alcohol



Scheme 13. Selected methods for the reduction of camphor to the corresponding secondary alcohol

results in efficiency above 90%. Furthermore, the effect of NH4Cl on the reaction course was examined; however, the alcohol yield was low because the mechanism led to the formation of amines [106].

Interesting methods for reducing camphor include the use of a) metals (Li, K) and ultrasound [107]; b) sodium sulfide nonahydrate in water at 42°C and UV-irradiation [108]. Scheme 13 shows the methodology for selective camphor reduction.

CONCLUSIONS

Some of the prominent methods of reducing ketones to secondary alcohols are the use of sodium borohydride or lithium aluminum hydride. However, these methods can be unreliable or lead to low yields. Many other methods are currently being developed: catalysts, enzymes, microwave techniques or ultrasounds. Such strategies in many cases allow for increasing the efficiency of the reaction, shortening its time or are an excellent pro-ecological alternative. The described methods of reducing selected ketones certainly cover such aspects as the use of renewable catalysts, the use of biodegradable materials or increasing energy efficiency, conducting chemical reactions in water or conducting solvent-free reactions. Considering the generally lower reactivity of ketones, it would be interesting to try to transfer these methods to aldehydes.

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