

Ruthenium(III) Chloride as an Efficient Catalyst for the Selective Oxidation of Fatty Alcohols to Aldehydes

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The oxidation of a series of fatty alcohols to yield the corresponding aldehydes is performed using $\text{RuCl}_3 \cdot (\text{H}_2\text{O})_x$ as a homogeneous catalyst and inexpensive trimethylamine *N*-oxide

(TMAO) as the oxygen source. The reaction conditions were optimized for both high conversion and high selectivity.

Introduction

Fatty alcohols are available from natural and thus sustainable sources for example through the hydrogenation of fatty acid esters (triglycerides).^[1] In nature, fatty alcohols are not only found in various fats and oils, but also in forestry waste and in particular in tall-oil, which is a side-product of the Kraft pulping process.^[2,3] Alkaline pulping of cellulose containing materials such as flax is another way to provide access to fatty alcohols.^[2,4] The oxidation products of these alcohols, in particular the corresponding fatty aldehydes, are of economic interest since the latter represent valuable starting materials and intermediates for chemical and pharmaceutical synthesis^[5] and have found wide applications in the flavors and fragrances industry.^[2,6]

Not surprisingly, the price of fatty aldehydes differs from the price of the corresponding fatty alcohols by 1–2 orders of magnitude.

In 2014, Cortés Corberán et al. summarized methods for the green oxidation of fatty alcohols in a review article.^[2] They pointed out that the “chemical reactivity of aliphatic alcohols is much lower than that of benzylic, allylic or functionalized alcohols”. In addition, the reactivity of fatty aldehydes for further oxidation is high under most reaction conditions. Therefore, it is still a challenging task to obtain high conversions as well as high selectivities.

Various routes for the selective oxidation of fatty alcohols to fatty aldehydes have been worked out in the past. These include enzymatic syntheses, as well as processes based on metal-containing catalysts^[2,7] and stoichiometric processes as the Swern or Moffat oxidation^[8] or oxidation with pyridinium chromate.^[9] A series of metals such as iron, zirconium,

palladium and ruthenium have been used as catalysts in these transformations as well as different oxidizing agents, among them iodosobenzene, hydroperoxides, hydrogen peroxide and oxygen.

Ruthenium has turned out to be of particular relevance for the selective generation of aldehydes from (long-chained) primary alcohols. Simple $\text{RuCl}_3 \cdot (\text{H}_2\text{O})_x$ in combination with peracids or hypochlorite, complexes such as $\text{RuCl}_2(\text{PPh}_3)_3$ with oxygen and/or TEMPO as well as a whole bunch of heterogeneous ruthenium-containing systems were applied in the past. Interestingly, these systems were mainly developed and optimized with 1-octanol as the substrate. The oxidation of typical members of the fatty alcohol series ($C \geq 12$) has rarely been reported.^[2,6]

Previously, we have published a study on the application of $\text{RuCl}_3 \cdot (\text{H}_2\text{O})_x$ as a highly active catalyst for the oxidation of various secondary and some primary alcohols with trimethylamine *N*-oxide (TMAO) as the oxidizing agent. This catalytic system provides the corresponding carbonyl derivatives with high selectivities and yields within a few minutes.^[7e] $\text{RuCl}_3 \cdot (\text{H}_2\text{O})_x$ is by far the cheapest source of ruthenium available. By DFT calculations on a structurally well-defined model complex, we were able to show that this reaction can be understood as a variant of the Ley-Griffith oxidation of alcohols, which normally uses perruthenate(VII) as the catalyst and mainly *N*-methylmorpholine *N*-oxide as the oxygen source. However, the history of ruthenium catalyzed alcohol oxidations dates back to 1976 when Sharpless et al. reported the use of a series of low-valent ruthenium compounds for the oxidation of alcohols to the corresponding carbonyl compounds.^[10]

Results and discussion

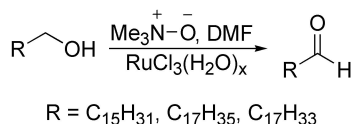
In the present manuscript we want to highlight the transfer of this simple, but largely efficient reaction system to long-chained primary alcohols, in particular to fatty alcohols such as hexadecanol, octadecanol and oleyl alcohol (Scheme 1).

In our recent study, this oxidation of alcohols was optimized in terms of temperature, reaction time, solvent and the amount and purity of the TMAO used. It soon turned out that the presence of water severely lowers the conversion rates of the catalytic alcohol oxidation. Accordingly, anhydrous TMAO and dry solvents were used in all reactions. Trimeth-

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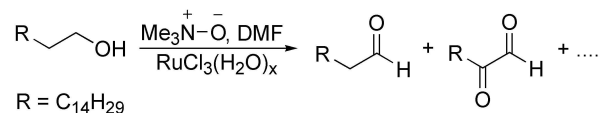


Scheme 1. Catalytic oxidation of primary alcohols.

ylamine *N*-oxide, which was purchased as the inexpensive dihydrate, was therefore dried before use by applying a Dean-Stark apparatus with toluene as the water-removing solvent.^[11] The water intake of 0.5 mol-% of hydrated RuCl₃ is negligible. The solvent dimethylformamide (DMF) was dried according to a procedure in the literature with CaSO₄ as the desiccant.^[12] 1,2-Dichlorobenzene was applied as the internal standard to quantify the conversions.

In advance to the broader study described below, a short series of experiments was carried out with 1-hexadecanol (palmityl alcohol) as the substrate to re-determine the optimum reaction conditions. Hereby, the temperature and the amount of the oxidant used were varied on the basis of our former results and the progress of the conversion and the yield of hexadecanal were investigated (Table 1).

Regardless of the temperature applied, the use of 3.0 equiv. of TMAO (Entries 2, 4 and 6) evidently leads to higher conversions and better yields than the use of 2.0 equiv. of TMAO (Entries 1, 3 and 5), indicating that there might be some deactivation of the oxidizing agent or the catalyst during the reaction. Even more interesting, after about one hour of reaction time, the formation of by-products seems to start to take place, since the yield of the aldehyde decreases while the conversion of 1-hexadecanol remains constant or still slightly increases. This is different to short reaction times, where the conversion rate reflects the aldehyde yield and is a clear indication that the aldehyde formed by the oxidation of the primary alcohol undergoes some subsequent transformations and is thus consumed again. The molecular structures of the resulting by-products could not be completely assigned yet. However, one singlet resonance in the ¹H NMR spectrum of the product mixture that appears close to the triplet signal of hexadecanal (9.61 ppm) at 9.38 ppm indicates that one of the



Scheme 2. Formation of 2-oxohexadecanal during the oxidation of 1-hexadecanol.

by-products could be 2-oxohexadecanal (Scheme 2, see the ¹H NMR spectrum in the Supporting Information). However, the formation of 2-oxohexadecanal alone does not explain the loss of yield of hexadecanal in total, since integration of the two aldehyde resonances does not sum up to the amount of converted 1-hexadecanol.

Typically for a reaction sequence of the type A→B→C, wherein the rate of the second step is slower than the rate of the first step, an optimum yield of B (here hexadecanal) can be obtained. A closer look to the entries of Table 1 reveals that at 60 °C more than 90% yield of hexadecanal are formed within 30 minutes, in case 3 equiv. of TMAO are applied (Entry 4). Thus, it is possible to achieve an almost complete conversion of the alcohol to the corresponding aldehyde. When the temperature is raised to 80 °C, an increase in by-product formation can be observed (Entry 6). As the temperature is lowered to 40 °C (Entry 2), the by-product concentration decreases, but the reaction rate also decreases rapidly leading to considerably lower conversions and yields. In summary, at 60 °C excellent conversion and yield are obtained at rather short reaction times. The formation of by-products is still slow enough to obtain the desired aldehyde purely. Accordingly, these reaction conditions were chosen for all following oxidation reactions.

In addition to hexadecanol, octadecanol and oleyl alcohol were oxidized with 2 equiv. of TMAO (Table 2, Entries 3 and 5) by applying the previously optimized reaction conditions. Comparing Entries 1 and 3 in Table 2 shows that the progress of conversion and yields is comparable for both, 1-hexadecanol and 1-octadecanol. This is in accordance with our expectations since both substrates only differ in the length of their carbon chains and are thus structurally rather similar. As already

Table 1. RuCl₃ catalysed oxidation of 1-hexadecanol at varying temperatures, utilizing different amounts of trimethylamine *N*-oxide (TMAO) as the oxidizing agent.

Entry ^[a]	T[°C]	Conversion ^[d] /Yield ^[d] (%) after				
		10 min	30 min	1 h	3 h	24 h
1 ^[b]	40	51/48	70/65	74/69	77/72	79/66
2 ^[c]	40	55/51	74/69	82/77	83/74	85/74
3 ^[b]	60	77/75	78/72	79/70	81/67	81/45
4 ^[c]	60	93/91	96/93	95/90	95/89	95/79
5 ^[b]	80	85/76	85/74	85/70	85/60	85/56
6 ^[c]	80	92/88	94/87	94/89	94/81	96/48

^[a] reaction conditions: substrate (1 mmol), anhydrous TMAO, 1 mL of dry DMF, 100 μL of 1,2-dichlorobenzene (internal standard), 0.5 mol-% of RuCl₃·(H₂O)_x;
^[b] 2.0 equiv. Of TMAO,
^[c] 3.0 equiv. Of TMAO,
^[d] conversion/yield determined by NMR spectroscopy.

Table 2. Substrate scope of the RuCl₃ catalysed oxidation of fatty alcohols with trimethylamine *N*-oxide.

Entry ^[a]	Substrate	Conversion ^[d] /Yield ^[d] [%] after				
		10 min	30 min	1 h	3 h	24 h
1	1-hexadecanol ^[b]	77/75	78/72	79/70	81/67	81/45
2	1-hexadecanol ^[c]	93/91	96/93	95/90	95/89	95/79
3	1-octadecanol ^[b]	81/78	82/76	83/74	82/68	85/53
4	1-octadecanol ^[c]	89/87	92/91	93/86	94/86	95/83
5	oleyl alcohol ^[b,e]	74/71	78/72	78/68	77/67	88/59
6	oleyl alcohol ^[c,e]	84/81	88/83	88/82	88/80	93/75
7	1-hexadecanol ^[c,f]	<1/<1	<1/<1	<1/<1	<1/<1	<1/<1

^[a] Reaction conditions: substrate (1 mmol), 1 mL dry DMF, 100 μ L 1,2-dichlorobenzene (internal standard), 0.5 mol-% of RuCl₃, 60 °C;
^[b] 2.0 equiv. of TMAO,
^[c] 3.0 equiv. of TMAO,
^[d] conversion/yield determined by NMR; ^[e] technical quality – consists of 53 % oleyl alcohol, 39 % linolyl alcohol and 8 % stearyl alcohol,
^[f] reaction in the absence of RuCl₃ - reaction conditions: substrate (1 mmol), 1 mL dry DMF, 100 μ L 1,2-dichlorobenzene (internal standard), 60 °C.

observed for hexadecanol, the increasing formation of by-products can also be observed at longer reaction times for octadecanol. The yield of octadecanal decreases by about 25 % between a reaction time of 10 min and 24 h, the yield of hexadecanal by about 30%. This again confirms that the aldehydes which are formed during the reaction undergo a variety of subsequent transformations. However, at short reaction times, octadecanal is formed without any visible by-products. The third substrate examined was oleyl alcohol (Table 2, Entry 5). This substrate was used in technical grade and therefore consists not only of oleyl alcohol (53 %) but also contains linolyl alcohol (39 %) and some octadecanol (9 %, stearyl alcohol). The composition of the substrate was determined using the ratios of typical resonances in the ¹H NMR spectrum (see the Supporting Information for further details). The reported conversions and yields refer to the entire mixture and not in particular to oleyl alcohol since all components generate a common ¹H NMR resonance of the corresponding fatty aldehyde (–C(O)H group). Due to the structural similarity of the three components in the substrate mixture, it can be assumed that the product composition reflects the substrate composition. Compared to hexadecanol and octadecanol, the oxidation of technical oleyl alcohol resulted in slightly lower yields. At a first glance, this might indicate that in addition to the alcohol function the double bonds being present in the unsaturated oleyl or linolyl alcohol could also undergo oxidation. However, no epoxide could be detected in the NMR spectra as there is no hint for any oxidation at the α -position to the C=C-double bonds.

Since the yields of aldehyde, which could be obtained using 2 equiv. of TMAO did not exceed 80 %, the oxidations of octadecanol and oleyl alcohol were also carried out with 3 equiv. of TMAO (Table 2, Entries 4 and 6). As already shown in Table 1 for hexadecanol (Entry 4), the increase of the amount of TMAO leads to a significant increase of conversions and yields during the first 30 minutes, which is also observed for the two new substrates examined (Table 2). The yield of 1-octadecanal increases by 15 % (Entry 4, 30 min reaction time) compared to the use of only 2 equiv. of TMAO (Entry 3, 30 min reaction

time). The conversion of oleyl alcohol (Entries 5 and 6) increases by 11 %.

Due to the exceptionally good yields, a scale-up of the reaction was carried out to allow the isolation of the product and to examine the possibility of performing the described reactions on a larger scale. For this purpose, the amounts of hexadecanol and solvent were increased by a factor of 40 as compared to Table 2, Entry 2. Hexadecanol, solvent and TMAO were heated to the reaction temperature, then the catalyst was added as stock solution in DMF. To ensure good yields, the reaction was stopped after 30 minutes. However, due to the high boiling point of DMF, it was not possible to isolate the product in a pure form. In addition, the excellent solubility of RuCl₃ in DMF prevented the separation of the catalyst using alumina or by extraction with both water and a series of organic solvents. These issues were motivation to look out for an alternative solvent. It finally turned out that acetone is a favourable solvent for these RuCl₃ catalyzed oxidations: The catalyst is readily soluble in it, it is easier to remove than DMF, more environmentally benign, available in large quantities and, finally, less expensive than DMF. Furthermore reaction temperatures of almost 60 °C are achievable. In acetone (Table 3, Entry 5) the reaction proceeds somewhat slower and the yield is reduced by about 5 %. Nevertheless, almost 90 % of the aldehyde can be obtained. The reaction was terminated after 3 h by cooling the mixture to r.t., acetone was removed under vacuum and diethyl ether was added, which allowed the simple separation of the catalyst and unreacted TMAO from the crude product by filtration through alumina. After removal of the diethyl ether, a crude product was isolated with 84 % yield, which consisted of 88 % of the aldehyde and 12 % of unreacted alcohol. The desired aldehyde could be purified by column chromatography, using *n*-hexane and dichloromethane (1:1) (the ¹H and ¹³C NMR spectra of the crude product mixture and the purified aldehyde are deposited in the Supporting Information).

Since changing the solvent to acetone offers a multitude of benefits a wider range of alcohols was oxidized in the presence of acetone as the solvent to investigate its suitability not only

Table 3. Substrate scope of the RuCl₃ catalysed oxidation of various alcohols using 3 equiv. of trimethylamine *N*-oxide and acetone as the solvent.

Entry ^[a]	Substrate	Conversion ^[c] /Yield ^[c] [%] after				
		10 min	30 min	1 h	3 h	24 h
1	1-phenylethanol ^[b]	99/99	99/99	99/99	99/99	99/99
2	cyclohexanol ^[b]	99/99	99/99	99/99	99/99	99/99
3	1-dodecanol ^[c]	81/78	86/86	86/83	88/84	89/68
4	1-hexadecanol ^[c]	79/76	84/82	85/83	91/88	90/37
5	1-octadecanol ^[c]	73/73	80/79	82/81	83/82	88/51
6	oleyl alcohol ^[c,d]	81/79	84/81	85/83	88/84	88/56

^[a] Reaction conditions: substrate (1 mmol), 1 mL dry acetone, 100 μL 1,2-dichlorobenzene (internal standard), 0.5 mol-% of RuCl₃, 55 °C;
^[b] turnovers/yields determined by gas chromatography;
^[c] conversion/yields determined by NMR;
^[d] technical substrate – consists of 53 % oleyl alcohol, 39 % linolyl alcohol and 8 % stearyl alcohol.

for the oxidation of 1-hexadecanol. The results are summarized in Table 3.

In summary, acetone was found to be nearly as good as a solvent as DMF. Secondary alcohols, both aromatic and aliphatic, are completely converted to the corresponding ketones within 10 min, as we have described in a previous publication for DMF.^[6] The conversion of primary alcohols, in particular of long-chained fatty alcohols, proceeds slightly slower in acetone compared to DMF. The obtained yields are decreased by about 2–10%. We believe that this can be explained by the decrease of the reaction temperature by 5 °C. Because ketones with a higher boiling point than acetone are in turn more difficult to remove from the reaction mixture and more expensive, they were not investigated further as solvents. In addition to the slower conversion of the alcohols, it was found that the formation of undesired by-products is also slowed down. The aldehyde yields increase during a period of 3 h and then start to decrease again. In DMF, the decomposition of the product starts after 30 min and then increases rapidly. Consequently, acetone allows a larger time frame for product isolation, which can be an advantage concerning the up-scaling of these oxidation processes.

Conclusion

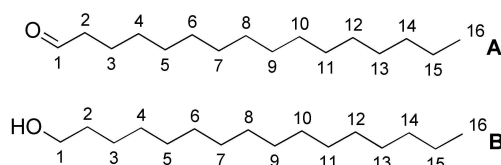
The results of the experiments discussed above demonstrate that it is possible to oxidize long-chain fatty alcohols efficiently to the corresponding aldehydes by using RuCl₃·(H₂O)_x as the catalyst and TMAO as the oxidizing agent in different polar and non-protic solvents. When 3 equiv. of TMAO are used, almost complete conversions in combination with high selectivities can be achieved with DMF as well as with acetone. Acetone provides a series of advantages over DMF, which are thoroughly discussed in the manuscript. To obtain high selectivities it is of particular importance to keep the reaction times low. At the moment we are investigating the formation of the 2-oxoaldehydes in more detail. These compounds are interesting starting materials, in particular for the synthesis of heterocyclic compounds, and they are difficult to obtain. It therefore is of interest to develop an efficient catalytic route to these compounds.

Experimental section

General procedure for catalytic oxidations: All catalytic experiments were performed in 20 mL crimp-capped vials supplied by VWR International GmbH. The vials were equipped with a magnetic stirring bar prior to weighing substrate and trimethylamin *N*-oxide (TMAO) and sealing the vial with a ptfte septum. After that, the vials were prepared for the reactions by three cycles of evacuation and flushing with nitrogen. Internal standard, solvent and catalyst (as DMF/acetone solution of defined concentration) were added using Hamilton syringes. The vials were placed in an aluminum block, which was preheated to the reaction temperature. Samples were taken using syringes and cannulas from B. Braun Melsungen AG and were then analyzed by NMR spectroscopy or gas chromatography. To separate the catalyst, the samples were sent over a small column of magnesium sulfate and neutral alumina.

Procedure for the large-scale oxidation of hexadecanol and the product isolation:

A 100 mL Schlenk tube was charged with 1-hexadecanol (9.90 g, 40 mmol) and TMAO (9.01 g, 120 mmol). The flask was prepared for the reaction by three cycles of evacuation and flushing with nitrogen. Dry acetone (40 mL) was added and the mixture was heated to 55 °C. RuCl₃(H₂O)_x (52.3 mg, 200 μmol, 0.5 mol-%) was dissolved in dry acetone (2 mL) and added to the solution. Hazard warning: *a large amount of gas is generated during the reaction. Therefore a vent for the gas must be provided, otherwise the reaction vessel may burst!* After approx. 3 h the solvent was removed under reduced pressure. Diethyl ether (50 mL) was added to the residue. The resulting suspension was passed over neutral alumina (column dimensions: l~20 cm, d~2 cm) to remove the catalyst and unreacted TMAO. The column was then rinsed with diethyl ether (200 mL). The solvent was removed from the filtrate to yield 8.09 mg (84 %) of the desired product. Composition of the crude product: 88 % of hexadecanal (A), 12 % of 1-hexadecanol (B) (Scheme 3). The aldehyde was purified by column chromatography using n-hexane and dichlormethane (1:1). 2.38 mg (9.88 mmol, 25 %) of a colorless solid were obtained. IR (KBr, cm⁻¹): $\tilde{\nu}$ 2951m, 2918s, 2847s, 2749w, 2712w, 1793w, 1732m, 1705m, 1635w, 1465m, 1411m, 1389m, 1373w, 1344w, 1300w, 1262w, 1241w, 1214w, 1197w, 1181w, 1127w, 1100w, 1067w, 1040w, 1024w, 981w, 894w, 867w, 802w, 716m, 697w, 659w, 605w, 578w, 545w, 518w, 508w, 464w, 453w, 432w. ¹H NMR (600.1 MHz, CDCl₃, 20 °C): δ 9.76 (t, ³J_{HH} = 1.8 Hz, 1H, A1), 2.41 (td, ³J_{HH} = 1.8 Hz, ³J_{HH} = 7.4 Hz, 2H, A2), 1.69–1.56 (m, 2H, A3), 1.36–1.17 (m, 24H, A4–A15), 0.87 (t, ³J_{HH} =



Scheme 3. Assignment of the NMR data of hexadecanal (A) and 1-hexadecanol (B)

7.0 Hz, 3H, A16). $^{13}\text{C}\{^1\text{H}\}$ NMR (150.9, CDCl_3 , 20 °C): δ 203.9 (A1), 44.1 (A2), 32.1 (A14), 29.8, 29.7, 29.6, 29.5 (A4–A12), 29.3 (A13), 22.8 (A15), 22.2 (A3), 14.3 (A16).

Supporting Information Summary

The Supporting Information contains further descriptions of preparative and catalytic procedures as well as all data of the chromatographic and spectroscopic characterization of the products.

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available in the supplementary material of this article.

Keywords: alcohol oxidation · catalysis · fatty aldehyde · ruthenium chloride · trimethylamine *N*-oxide

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