Journal of Organometallic Chemistry 701 (2012) 51–55

Contents lists available at SciVerse ScienceDirect

Journal of Organometallic Chemistry

journal homepage: www.elsevier.com/locate/jorganchem

# Halide substituted Schiff-bases: Different activities in methyltrioxorhenium(VII) catalyzed epoxidation via different substitution patterns

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ARTICLE INFO

*Article history:*
Received 11 November 2011
Received in revised form
5 December 2011
Accepted 7 December 2011

*Keywords:*
Epoxidation
Homogeneous catalysis
Methyltrioxorhenium (MTO)
Schiff-base
Hydrogen peroxide

ABSTRACT

This report shows the influence of halide substituted Schiff-bases as ligands of methyltrioxorhenium (MTO) in epoxidation catalysis. Therefore, selected Schiff-bases were prepared by the reaction of hydroxy-benzaldehydes and aniline derivates. These differently substituted Schiff-bases were tested as MTO-ligands in cyclooctene- and 1-octene-epoxidation. Although no great disparities among the substitution patterns have been found, some conclusions can be drawn. Flourines are inferior to chlorines or bromines as substituents. Halides in *ortho*-position lead to higher activities than in *para*- or *meta*-position. The balance between electron donating and withdrawing influences at the Schiff-base plays a prominent role in their utility as ligand to MTO in epoxidation catalysis.

## 1. Introduction

Methyltrioxorhenium (MTO) has proven to be a highly versatile catalyst for a plethora of organic reactions [1–5]. Best examined is olefin oxidation [2,3,6–33]. The particular advantage of MTO is its compatibility with hydrogen peroxide as oxidizing agent, leading to water as the only byproduct. However, diol formation in the presence of Lewis acidic MTO and catalyst decomposition in aqueous solutions are major problems [34,35]. When trying to overcome both problems it was found that the presence of Lewis-bases in significant excess stabilizes MTO, accelerates the oxidation reaction and leads to significant diol reduction [16,22,25,27,36–39]. It was further observed that Schiff-bases as additives need not to be applied in excess to reach similar effects [21,26,30,32,33,40–42]. However, it has also been shown that the scope of the Schiff-base supported epoxidation catalyzed by MTO appears to have a more limited substrate scope than the reaction supported by aromatic Lewis-bases [20,43,44]. Furthermore, the so far published results are not all matching well.

In this work the influence of different electron withdrawing groups, especially halides at different positions is examined. Two different substrates, cyclooctene and 1-octene have been chosen. Cyclooctene was selected because of its widespread use in former studies. Many scientific reports are available based on this substrate to compare the obtained results with respect to the general activity of the catalytic system. 1-octene is chosen since its oxidation is much slower than that of cyclooctene and comparison of the two substrates should reveal the influence of the different substituents quite well.

## 2. Experimental

### 2.1. Catalysis

Method A: 441 mg cyclooctene (4 mmol, 0.52 mL), 0.5 mL mesitylene (internal standard), 1 mol% catalyst (0.04 mmol MTO + 0.04 mmol Schiff-base), 2 mL nitromethane ($MeNO_2$).

Method B: 449 mg 1-octene (4 mmol, 0.63 mL), 0.2 mL mesitylene (internal standard), 0.2 mL toluene (internal standard), 1 mol% catalyst (0.04 mmol MTO + 0.04 mmol Schiff-base), 2 mL nitromethane.

The compounds from methods A and B were combined in tempered glas vessles and vigorously stirred. The reaction was started by addition of 0.91 mL aqueous $H_2O_2$ solution (27%, 8 mmol). Samples of 0.2 mL were taken after 5 min, 30 min and 1 h. Samples were treated with $MnO_2$ to destroy unreacted hydrogen peroxide and $MgSO_4$ to remove water. They were subsequently filtered and afterwards diluted with dichloromethane (DCM) for GC analysis. Analysis was carried out by calibrated GC methods in all cases.

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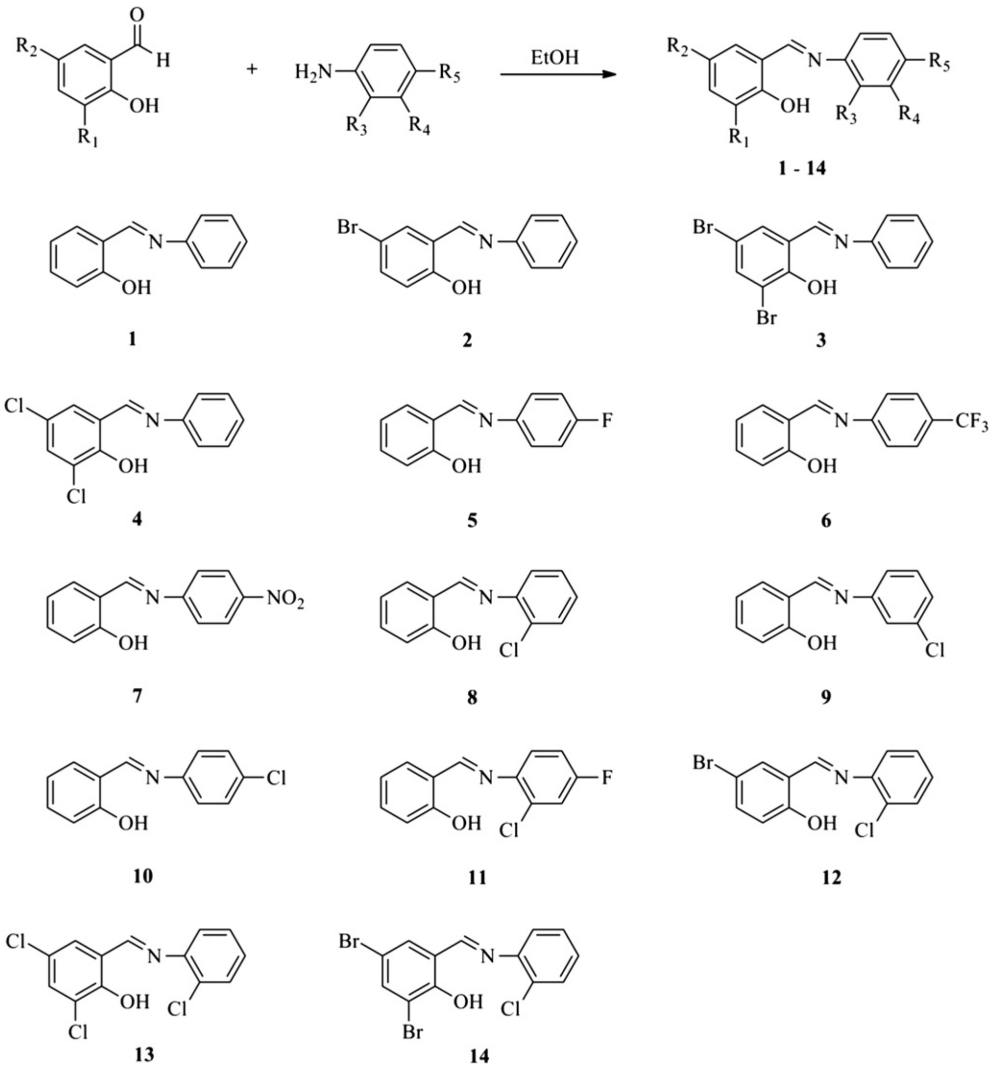
0022-328X/$ – see front matter © 2011 Published by Elsevier B.V.
doi:10.1016/j.jorganchem.2011.12.010



**Scheme 1.** Reaction of hydroxy-benzaldehydes and aniline derivates to Schiff-bases.

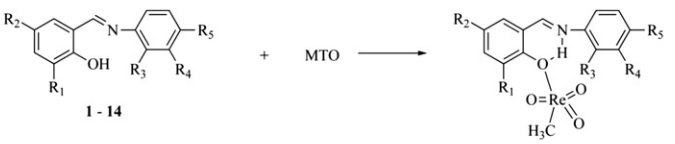
### 2.2. *General procedure for the synthesis of ligands*

A solution of salicylaldehyde derivative (1.0 equiv.) in ethanol (5.0 mL mmol$^{-1}$) was added into a solution of aniline derivative (1.0 equiv.) in ethanol (5.0 mL mmol$^{-1}$). After stirring at room temperature for 1 h, the mixture was refluxed until complete consumption of the starting materials. Subsequently, ethanol was removed under reduced pressure and the obtained imines were purified by crystallization. The obtained Schiff-bases were characterized by NMR and elemental analysis. All analytical data is in accordance with literature [21,26,30,32,33,43].

## 3. Results and discussion

A set of substituted Schiff-bases was synthesized (Scheme 1). All Schiff-bases were obtained by reacting different hydroxy-benzaldehydes with aniline derivates in ethanol according to published procedures [21,26,30,32,33,43]. Based on former research results, indicating that electron donating groups decrease the catalyst activity and reduce the lifetime of the catalyst, electron withdrawing groups, especially halides were chosen for this study.

All obtained Schiff-bases react with MTO to form stable complexes (Scheme 2). In accordance with earlier examinations the $^1$H NMR data show a high field shift of the methyl group of MTO adducts of **1**–**14** compared to free MTO (Table 1). This observations



**Scheme 2.** Formation of MTO-Schiff-base complex.

**Table 1**
Selected $^1$H NMR spectroscopic data for MTO-Schiff-base complexes in $CDCl_3$.

| Compound | δ($^1$H) MTO-$CH_3$ | Compound | δ($^1$H) MTO-$CH_3$ |
|---|---|---|---|
| MTO | 2.68 | +**7** | 2.62 |
| +**2** | 2.60 | +**11** | 2.62 |
| +**4** | 2.62 | +**12** | 2.62 |
| +**5** | 2.62 | +**13** | 2.62 |
| +**6** | 2.62 | | |

**Table 2**
Cyclooctene-epoxidation yields and TOFs[a] catalyzed by MTO.

| Entry | Ligand | MTO [mol%] | Solvent | Yield 30 min [%] | TOF [$h^{-1}$] |
|---|---|---|---|---|---|
| 1 | – | 1 | $MeNO_2$ | 100 | 840 |
| 2 | **1** | 1 | $MeNO_2$ | 78 | 650 |
| 3 | **2** | 1 | $MeNO_2$ | 99 | 790 |
| 4 | **3** | 1 | $MeNO_2$ | 100 | 840 |
| 5 | **4** | 1 | $MeNO_2$ | 99 | 750 |
| 6 | **5** | 1 | $MeNO_2$ | 99 | 720 |
| 7 | **6** | 1 | $MeNO_2$ | 99 | 760 |
| 8 | **7** | 1 | $MeNO_2$ | 88 | 660 |
| 9 | **8** | 1 | $MeNO_2$ | 100 | 700 |
| 10 | **9** | 1 | $MeNO_2$ | 91 | 670 |
| 11 | **10** | 1 | $MeNO_2$ | 99 | 740 |
| 12 | **12** | 1 | $MeNO_2$ | 100 | 770 |
| 13 | **13** | 1 | $MeNO_2$ | 96 | 770 |
| 14 | **14** | 1 | $MeNO_2$ | 92 | 710 |
| 15 | $^{t}$butylpyridine (1:5)[b] | 1 | $MeNO_2$ | 100 | 1100 |
| 16 | **8** | 0.1 | $MeNO_2$ | 39 | 2600 |
| 17 | **9** | 0.1 | $MeNO_2$ | 33 | 2100 |
| 18 | **10** | 0.1 | $MeNO_2$ | 33 | 2100 |
| 19 | **2** | 1 | DCM | 90 | 670 |
| 20 | **3** | 1 | DCM | 83 | 490 |

[a] TOF calculated after 5 min: [(mol epoxide)/(mol catalyst *h)].
[b] ratio MTO:ligand.

proof the tethering of the Schiff-base to MTO [32,33]. Among the Schiff-bases-adducts no significant difference between halide or nitro (**7**) substitution can be observed.

To rule out solvent molecules as ligands to MTO instead of Schiff-bases we chose nitromethane ($MeNO_2$) as a very weak coordinating solvent for the catalysis tests [43]. Furthermore, blank experiments without catalyst show no formation of epoxide neither with cyclooctene nor with 1-octene.

For the first part of the catalysis examination, cyclooctene was applied as substrate, as it is a very active starting material, leading to a comparatively stable epoxide. Based on earlier experiments, MTO and the Schiff-base-ligands have been used in a ratio of 1:1 for this study [21,32,33]. The catalyst:substrate:oxidant ratio was chosen to be 1:100:200 and the substrate concentration in the reaction solution was 1 M. Hydrogen peroxide (aqueous solution 27%) was used as oxidant. All of the ligands examined with MTO show similar results (Table 2). The turn-over-frequencies (TOFs) are ranging between 700 $h^{-1}$ and 800 $h^{-1}$ and the yield is quantitative after 30 min reaction time in almost every case. Only the non-substituted Schiff-base **1** as well as the nitro-substituted Schiff-base **7** are worse, reaching TOFs of around 650 $h^{-1}$ and yields of 78% and 88%, respectively (Table 2, entries 2 and 8). As indicated in earlier examinations, a reason for that might be the relatively good donor ability of unsubstituted Schiff-base **1** compared to the halide substituted Schiff-bases [33]. On the other hand, the nitro-group of Schiff-base **7**, as very strong electron acceptor, seems to withdraw too much electron density from the Schiff-base-system to operate the catalysis satisfactorily. The catalytic performance of MTO-Schiff-base adducts relies most likely on an equilibrium of electron donating and electron accepting effects. If the equilibrium is shifted too much towards one side, the activity of the catalyst decreases.

Chlorine and bromine substituents lead to similar results (Table 2, entries 3–5, 9–11). The performance of the fluorine-substituted compounds **5** and **6** is slightly lower (Table 2, entries 6 and 7). The position of the halide has no visible influence on the fast cyclooctene-epoxidation (Table 2, entries 9–11). It does also not matter if the halide is located at the hydroxy-benzaldehyde moiety or at the aniline moiety (Table 2, entries 3–5, 9–11). Substituting both aromatics does also not increase the catalyst's activity (Table 2, entries 12–14).

Lower catalyst concentrations of 0.1 mol% give 40% yield after 30 min with a TOF of 2600 $h^{-1}$ for **8** as ligand of MTO. When comparing to an earlier study of Sharpless et al. who also worked with nitromethane as solvent, activities are quite similar. Using MTO and pyridine in a ratio of 1:1 leads to 40% yield after 5 min in the experiments performed by Sharpless et al. A 70% yield is obtained after 5 min with pure MTO and doubled catalyst concentration as applied in ref. 16. Experiments with 4-*tert*-butylpyridine (1:5) yielded 95% product after 5 min whereas 90% yield are reported for the MTO-pyridine ratio of 1:12 [27].

Compared to other experiments with Schiff-bases, carried out earlier in our group, the results in nitromethane are much better than those in dichloromethane or without solvent (Table 4) [21,32,33]. A yield of 78% is obtained with Schiff-base **1** after 30 min compared to 4 h in earlier studies without solvent, which equals to an acceleration by factor eight [33]. When the Schiff-base is replaced by a 4-*tert*-butylpyridine ligand, the expected increase in activity can be observed. After 5 min a yield of 95% and a TOF of 1100 $h^{-1}$ are reached (Table 2, entry 15). Changing the solvent from nitromethane to dichloromethane results in a slightly lower activity (Table 2, entries 19, 20 vs. entries 3, 4). Comparing the nitromethane-experiments to earlier studies nitromethane seems to be a better or at least an equal solvent if the different experimental setups are taken into account (Table 4).

Using a terminal alkene like 1-octene as substrate leads to similar results as obtained with cyclooctene with respect to the differences between pure MTO and MTO plus Schiff-base. The yield after 3 h ranges between 61% and 74% and the TOFs vary from 70 $h^{-1}$–80 $h^{-1}$ (Table 3). All examined systems are almost equal except nitro-substituted compound **7** (56% yield, 50 $h^{-1}$) (Table 3, entry 8). The reasons are most likely the same as described for cyclooctene. Again, using nitromethane as solvent shows very good results compared to other systems working with 1-octene (Table 4). Slight differences occur between chlorine-substituted ligands leading to an activity order $o > p > m$ (Table 3, entries 9–11). The highest activity can be achieved with the trichloro-substituted compound **13** (74% yield). Interestingly, with 4-*tert*-butylpyridine as additive the catalyst system decomposes rapidly. In the beginning it is more active indicated by the higher TOF of 100 $h^{-1}$ but after 3 h the yield remains at 31%, whereas the stability of the Schiff-base supported catalyst lasts longer and higher conversions

**Table 3**
1-Octene epoxidation yields and TOFs[a] catalyzed by MTO.

| Entry | Ligand | MTO [mol%] | Solvent | Yield 3 h [%] | TOF [$h^{-1}$] |
|---|---|---|---|---|---|
| 1 | – | 1 | $MeNO_2$ | 69 | 70 |
| 2 | **1** | 1 | $MeNO_2$ | 61 | 70 |
| 3 | **2** | 1 | $MeNO_2$ | 72 | 70 |
| 4 | **3** | 1 | $MeNO_2$ | 72 | 70 |
| 5 | **4** | 1 | $MeNO_2$ | 69 | 70 |
| 6 | **5** | 1 | $MeNO_2$ | 62 | 80 |
| 7 | **6** | 1 | $MeNO_2$ | 69 | 70 |
| 8 | **7** | 1 | $MeNO_2$ | 56 | 50 |
| 9 | **8** | 1 | $MeNO_2$ | 72 | 70 |
| 10 | **9** | 1 | $MeNO_2$ | 67 | 70 |
| 11 | **10** | 1 | $MeNO_2$ | 69 | 70 |
| 12 | **11** | 1 | $MeNO_2$ | 66 | 80 |
| 13 | **12** | 1 | $MeNO_2$ | 73 | 80 |
| 14 | **13** | 1 | $MeNO_2$ | 74 | 80 |
| 15 | **14** | 1 | $MeNO_2$ | 67 | 70 |
| 16 | $^{t}$butylpyridine (1:5)[b] | 1 | $MeNO_2$ | 31 | 100 |

[a] TOF calculated after 5 min: [(mol epoxide)/(mol catalyst *h)].
[b] ratio MTO:ligand.

**Table 4**
Collected data concerning MTO catalyzed epoxidation of cyclooctene and 1-octene.

| Ref | Ligand | Substrate *c* [mol/l][a] | MTO [mol%] | Solvent | Reaction time [h] | Yield [%] |
|---|---|---|---|---|---|---|
| [27] | pyridine | cyclooctene<br>0.8 | 0.5 | $MeNO_2$ | 0.5 | 95 |
| [27] | pyridine | cyclooctene<br>2.0 | 0.5 | DCM | 2 | 99 |
| [16] | 3-cyanopyridine | cyclooctene<br>2.3 | 1 | – | 4 | 75 |
| [33] | **1** | cyclooctene<br>2.0 | 1 | – | 4 | 70 |
| [33] | **10** | cyclootene<br>2.0 | 1 | – | 4 | 79 |
| [38] | 4,4′-dimethyl-2,2′-bipyridine | cyclooctene<br>0.9 | 1 | DCM | 4 | 78 |
| [37] | pyridine | cyclootene<br>1.3 | 0.5 | $CHCl_3$ | 0.5 | 79 |
| [45] | 3-methylpyrazole | cyclootene<br>1.1 | 0.1 | DCM | 2 | $>$ 99 |
| [17] | 3-methylpyrazole | cyclootene<br>3.5 | 0.1 | – | 3 | $>$ 99 |
| [46] | pyrazole | cyclooctene<br>1.2 | 0.1 | trifluoroethanol | 1 | 100 |
| [21] | Schiff-base | cyclooctene<br>0.2 | 1 | DCM | 4 | 70 |
| [20] | 4-*tert*-butylpyridine | cyclooctene<br>0.7 | 1 | *tert*-butanol | 0.5 | 78 |
| [23] | pyrazole | cyclooctene<br>0.8 | 0.5 | DCM | 2 | 96 |
| [23] | pyridine | cyclooctene<br>0.8 | 0.5 | DCM | 2 | 96 |
| [45] | 3-methylpyrazole | 1-octene<br>1.1 | 0.5 | DCM | 8 | 91 |
| [17] | 3-methylpyrazole | 1-octene<br>3.5 | 0.5 | – | 8 | 62 |
| [21] | Schiff-base | 1-octene<br>0.2 | 1 | DCM | 24 | 100 |
| [46] | pyrazole | 1-octene<br>1.2 | 0.1 | trifluoroethanol | 21 | 100 |

[a] If not given directly, calculated from experimental sections.

are reached. For an equal stabilization a higher amount of pyridine is needed for the MTO/pyridine catalyst system.

## 4. Conclusion

Several halide substituted Schiff-bases, have been examined in epoxidation for cyclooctene- and 1-octene-epoxidation in the presence of MTO and with $H_2O_2$ as oxidizing agent. The addition of Schiff-bases has not a pronounced influence on the catalytic activity and selectivity. In some cases a small increase of yield and TOF is observed if Schiff-bases are added to the reaction solution (Tables 2, 3). Fluorine substitution (**5**, **11**) leads to slightly lower yields than substitution with other halides. No significant differences occur when chlorine and bromine moieties are applied. The yields of such systems range between 67% and 74% after 5 min reaction time. It also has no significant influence whether the hydroxy-benzaldehyde or the aniline moiety or both of them are substituted. The *ortho*-, *meta*-, *para*-substitution at the aniline moiety shows a reaction order of $o > p > m$. The exchange of Schiff-bases as ligands by 4-*tert*-butylpyridine leads to a higher conversion and turn-over-frequency if cyclooctene is used as substrate. The epoxidation of 1-octene displays a higher activity only early in the reaction. The overall yield after 3 h is lower than that of MTO plus Schiff-bases. The catalytic optimum of MTO-Schiff-base-systems is based on fine tuning the electron donating and the electron accepting character of the Schiff-base. The utilization of nitromethane as solvent for the catalysis turned out to be much more rewarding than catalysis with other or without solvents.

## Acknowledgment

PA thanks TUM-GS for financial support.

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