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Interaction of dihydropyridines with alkoxycarbene complexes of tungsten: formation and use as cyclopropanation reagents of pyridinium ylide complexes

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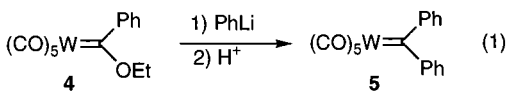
Abstract

Alkoxycarbene complexes of tungsten, e.g. $(CO)_5W{=}C(CH_3)OEt$ (**13**) react with 1,2- and 1,4-dihydropyridines, to give, upon transfer of a hydride to the carbene carbon, elimination of ethanol, and readdition of pyridine to an unstable alkylidene complex $(CO)_5W{=}C(H)CH_3$ (**22**), pyridinium ylide complexes of the type $(CO)_5W^- - C(H)(CH3)Py^+$ (**17**). A second pyridinium ylide complex $(CO)_5W-C(H)(CH_3)C_5H_7N$ (**16**), and a third dihydropyridinium complex $(CO)_5W-C(H)(CH_3)C_8H_{11}N$ (**15**), resulting from the interaction of respectively 2,5-dihydropyridine and 5-isopropylidene-2,5-dihydropyridine with $(CO)_5W{=}C(H)(CH_3)$ were also isolated and fully characterized by X-ray crystallography. The origin of the two latter complexes could be established. The new *N*-ylide complexes react with olefins to give cyclopropanes. Especially interesting, as far as the yields are concerned, is the interaction of highly nucleophilic olefins such as enamines with $(CO)_5W^- - C(H)PhPy^+$ (**25**) which leads to isomeric cyclopropylamines. The mechanism of their formation will be discussed. © 1998 Elsevier Science S.A. All rights reserved.

Keywords: Alkoxy carbenes; Tungsten; Dihydropyridines; Pyridinium ylide complexes; Cyclopropanations

1. Introduction

A way to stabilize organic carbenes **1** (Scheme 1) and to allow their detection by physical methods, especially by UV spectroscopy, is to react them, at low temperature with pyridine [1]. This interaction leads to pyridinium ylides **3** the lifetime of which is much longer than that of the starting carbenes. In contrast to most organic carbenes, Fischer-type carbene complexes are stable species, which can be easily handled at room temperature (r.t.) [2]. Moreover, several alkylidene complexes of tungsten and chromium could also be prepared: thus diphenylethylidene complex of tungsten **5** was obtained via the reaction of the corresponding phenyl ethoxy carbene complex **4** with PhLi, followed by an acid treatment [3], and the benzylidene tungsten complex **7** was prepared both by Casey and Fischer [4,5], by borohydride reduction of the corresponding phenyl ethoxy carbene complex **4**.



4 **5** (1)

However, both alkylidene complexes are less stable than the corresponding starting complex. Whereas the former could be handled at r.t., the latter was only stable at low temperature. Stabilization of both complexes could however be achieved by their reaction with triphenylphosphine, which gave the corresponding phosphonium ylides (e.g. **7**→**8**) [4].

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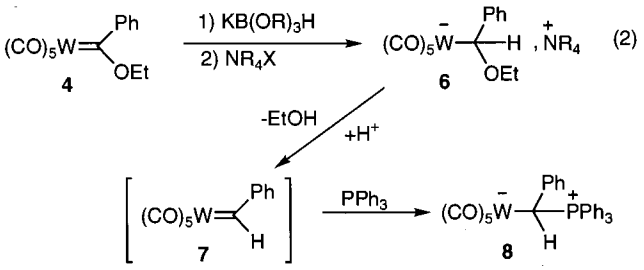


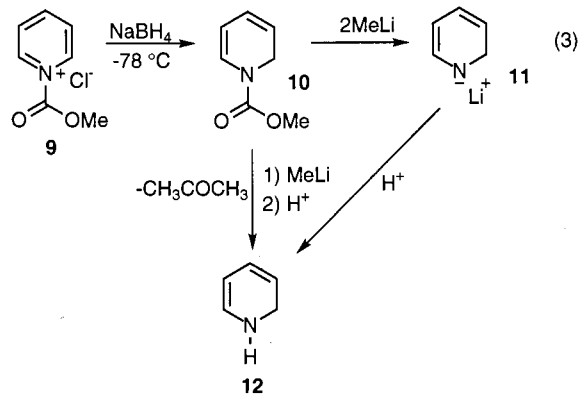
The purpose of this paper is to describe a new general method for the preparation and stabilization of alkylidene complexes of tungsten(0), as pyridinium ylide complexes, and to demonstrate that these complexes can be used as synthons for the cyclopropanation of olefins.

2. Results and discussion

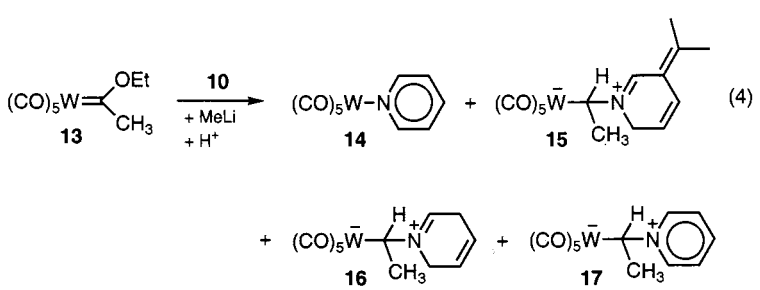
Alkoxycarbene complexes of the Fischer-type can be compared, as far as some of their reactions are concerned, to carbonyl derivatives, e.g. ketones or esters. Two typical transformations which confirm this analogy are the aminolysis of alkoxycarbene complexes which leads to aminocarbene complexes [6], the analogs of amides, and their reduction with hydrides, which gives rise to tungstates $(CO)_5W^-–C(H)(OR)(R)\ M^+$ [4].

It is known that carbonyl compounds can be reduced by dihydropyridines to alcohols, a reaction which is well documented in biochemistry (Scheme 2). In order to further the analogy between carbene complexes and carbonyl compounds, we explored the possibility of using dihydropyridines as reducing agents for alkoxycarbene complexes.

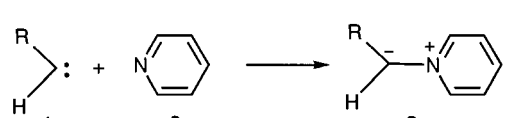
According to the literature, protected 1,2-dihydropyridine **10** is easily prepared as a single isomer, by low temperature reduction of pyridinium chloroformate **9** with $NaBH_4$ [7]. Removal of the protecting group is achieved by treatment with excess MeLi, followed by protonation.



Thus, when 1,2-dihydropyridine **12**, prepared by this procedure, was added at 0°C to an ethereal solution of $(CO)_5W{=}C(OEt)CH_3$ (**13**), a change of the colour from yellow to orange rapidly took place [8]. After 1 h, most of the starting complex had reacted, and four new complexes could be detected by TLC, as difficult to separate pairs of products. Careful silica gel chromatography allowed nevertheless the separation and characterization of the new complexes. The less polar complex was given structure **14**, the known (pentacarbonyl) tungsten pyridine complex [9]. The second complex, isolated in a 8.5% yield, agreed with structure **15**, a 5-isopropylidene-2,5-dihydropyridinium ylide complex of tungsten, on the grounds of its spectroscopic data (Table 1), and confirmed by X-ray spectroscopy (Fig. 1).



The third complex, isolated in a 20% yield, had structure **16**, again a complex of a 2,5-dihydropyridine as shown by its X-ray structure on Fig. 2. Finally, to the most polar complex, the main product of the reaction (50%) was given structure **17**, the ethylidene pyridinium ylide of tungsten pentacarbonyl. Its ORTEP view is shown on Fig. 3. The most important bond distances and bond angles of these three complexes are shown in Table 1.



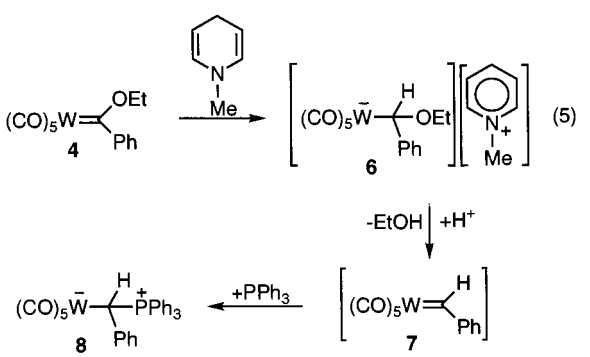
Scheme 1.

Table 1
^{1}H and ^{13}C-NMR data, crystallographic data and bond distances (Å) and angles (°) for complexes **15**, **16** and **17**

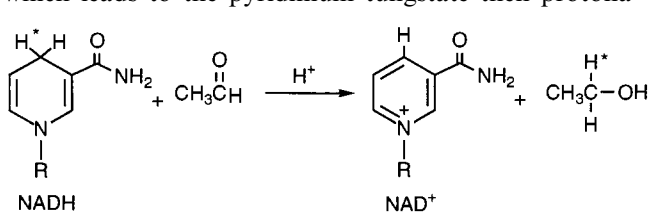
Complex	NMR data		X-ray data				
	δ ^{1}H $H(1)$	δ ^{13}C $C(1)$	W–C(1)	C(1)–C(2)	C(1)–N(1)	W–C(1)–C(2)	W–C(1)–N(1)
15	3.86	52.6	2.381(9)	1.51(1)	1.45(1)	113.2(6)	114.1(5)
16	3.81	54.1	2.34(2)	1.48(2)	1.54(2)	116.6(13)	109.2(11)
17	4.90	57.2	2.32(2)	1.35(3)	1.49(2)	122.8(16)	112.3(9)

2.1. Mechanism of the reduction reaction. Origin of the new pyridinium ylide complexes

If the interaction of dihydropyridine with carbene complexes follows the same pathway as with carbonyl compounds, hydride transfer to the carbene carbon should be observed. Although hydrogen is present on the former carbene carbon of the three new complexes, the course of the reduction reaction is more complicated. In order to gain more insight into the new reduction process, interaction of alkoxycarbene complex 4 with *N*-methyl-1,4-dihydropyridine [10], which is also a reducing dihydropyridine was examined: in that case a pyridinium tungstate **6** (N^+R_4=$MePy^+$) was isolated and characterized by NMR spectroscopy. Treatment of **6** with CF_3CO_2H led to the expected benzylidene complex **7** which could be stabilized as its known triphenylphosphonium ylide **8** and fully characterized. Thus, hydride transfer to the carbene carbon takes indeed place; moreover, protonation by CF_3CO_2H at the oxygen atom of the ethoxy group induced the elimination of ethanol and the formation of the benzylidene complex



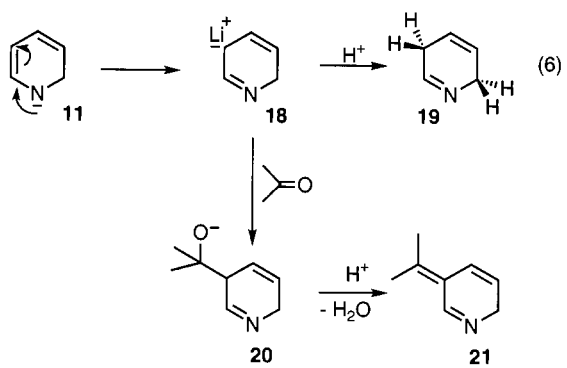
The same set of reactions could thus take place in the case of 1,2-dihydropyridine: first, a hydride transfer which leads to the pyridinium tungstate then protonation by pyridinium at oxygen followed by elimination of ethanol and formation of the benzylidene complex **7** and pyridine. Interaction of the two latter species would then lead to the observed pyridinium ylide complex Scheme 3.



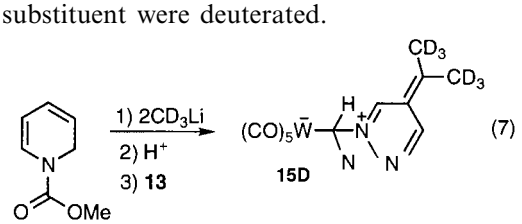
Scheme 2.

2.2. Formation of 2,5-dihydropyridinium ylide complexes

The fact that complexes **15** and **16** had the same origin, complex $(CO)_5W{=}C(H)CH_3$ (**22**) and the lithium amide of 1,2-dihydropyridine **11**, was demonstrated in the following way. The interaction of the protected dihydropyridine **10** with two equivalents of MeLi leads to the lithium amide **11** and acetone. Protonation of **11** leads to 1,2-dihydropyridine **12** (Eq. 3). However, **11** can be in equilibrium with the carbon–lithio compound **18**: upon protonation **18** could indeed lead to 2,5-dihydropyridine **19**. Moreover, reaction of **18** with acetone might also take place and lead, after dehydration to 5-isopropylidene-2,5-dihydropyridine **21**.



Confirmation of this hypothesis was obtained by the following simple reaction: addition of two equivalents of CD_3Li to the protected 1,2-dihydropyridine followed by protonation, and reaction with complex **13** led inter-alia to complex **15D**, in which, according to ^{1}H and ^{13}C-NMR spectra both methyl groups of the isopropylidene substituent were deuterated.



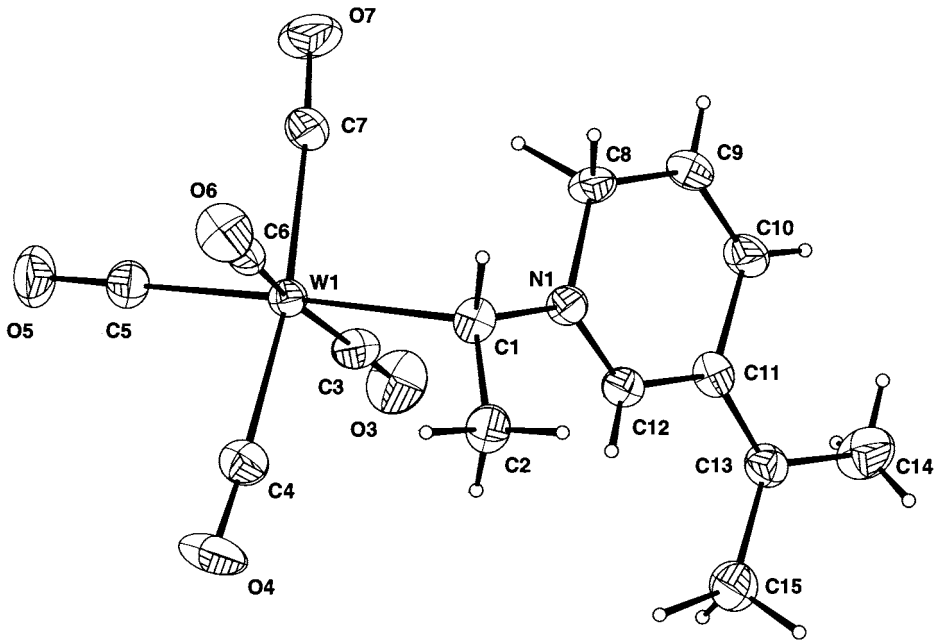
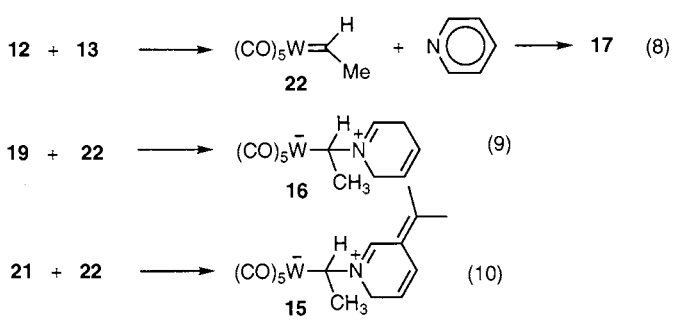


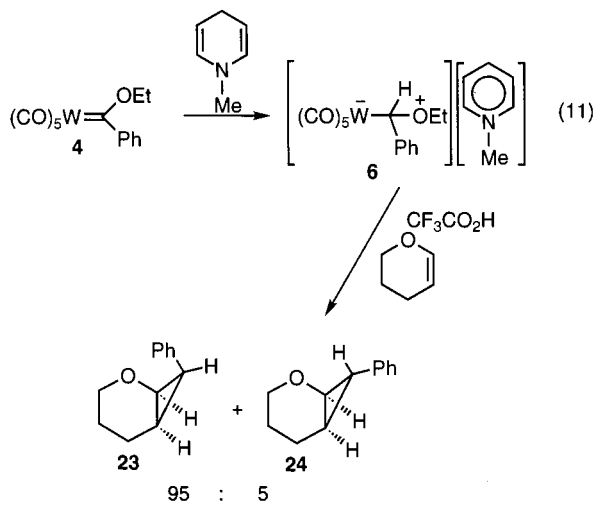
Fig. 1. CAMERON projection of complex **15** with the atomic numbering scheme.

Thus, 1,2-dihydropyridine 'reduces' the alkoxy carbene complex **13** to the ethylidene complex **22** with formation of pyridine. The reaction of **22** with pyridine gives the pyridinium ylide complex **17**, the main product of the reaction. The reaction of **22** with the more stable (less oxidable) 2,5-dihydropyridine **19** and 5-isopropylidene-2,5-dihydropyridine **21** leads, respectively to the dihydropyridinium complexes **16** and **15**. Excess of MeLi reacts with acetone and suppresses the formation of complex **15**.



*2.3. Pyridinium tungstate **6** as a cyclopropanation reagent*

The interaction of the pyridinium tungstate **6** with CF_3CO_2H, in the presence of triphenylphosphine led to the phosphonium ylide complex **8**, providing strong evidence for the formation of the benzylidene complex **7**. This could also be confirmed by its capture with dihydropyrane. Thus, the reaction of *N*-methyl dihydropyridine with complex **1** at −78°C led to a dark–red solution of the pyridinium tungstate **6**. After evaporation of the solvent and successive additions of excess dihydropyrane and CF_3CO_2H, a mixture of *cis* and *trans* cyclopropane derivatives was formed and isolated in 35% yield. Confirmation of their structure and stereochemistry was secured by ^1H and ^{13}C-NMR and by NOE experiments.



2.4. Pyridinium ylide complexes of tungsten as cyclopropanation reagents for enamines

In a previous publication [8], the transfer of the alkylidene moieties of the pyridinium ylide complexes to olefins was described. Especially rewarding were the intramolecular reactions, and also the intermolecular reactions involving electron-rich olefins such as enol ethers. It was our feeling that enamines might be the olefins of choice for successful intermolecular cyclopropanation reactions. And this was indeed the case. A

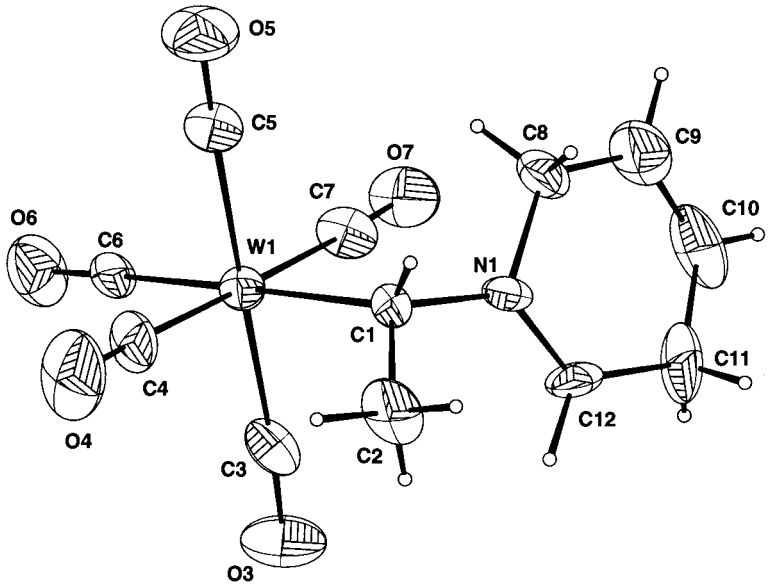


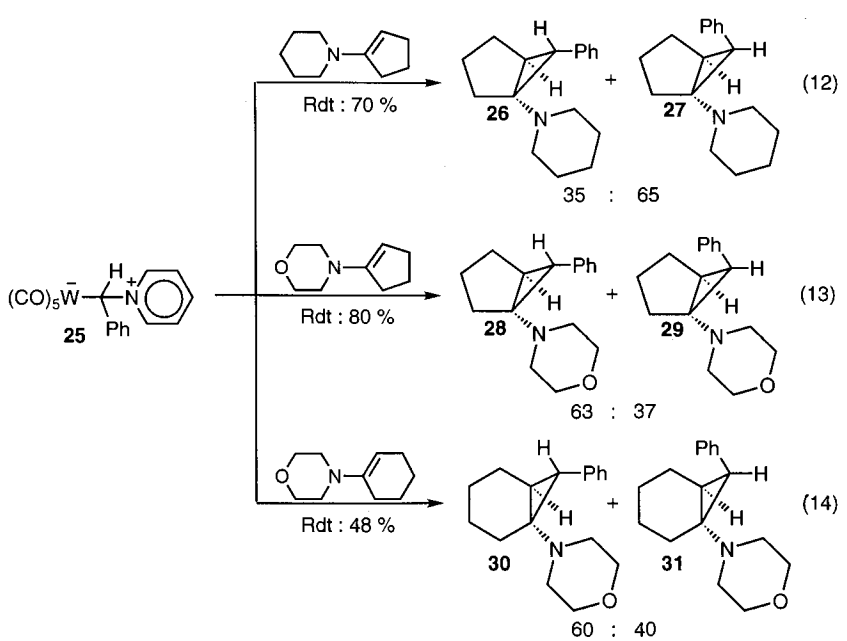
Fig. 2. CAMERON projection of complex **16** with the atomic numbering scheme.

series of enamines [11], derived from cyclopentanone and cyclohexanone, reacted with complex **25** at r.t. and within the space of 1 h, to give high yields of the corresponding cyclopropylamines as mixtures of isomers. These were easily separated by silica gel chromatrography. To the less polar products were given structures **26**, **28** and **30** on the grounds of NMR experiments, whereas **27**, **29** and **31** had the opposite stereochemistry. Cyclopropylamines had already been obtained from enamines and precursors of carbenes by other methods: however, transfer of the benzylidene carbene has to the best of our knowledge not been described [12].

As far as the mechanism of these cyclopropanation reactions is concerned, it is likely that enamines might easily be able to displace the pyridine from the ylides, with concomitant formation of a carbon–carbon bond, pyridinium ylide complexes being here considered as alkylating agents (Scheme 4). Reaction of the negatively charged tungsten atom with the carbon of the intermediate iminium group, might then lead to a tungstacyclobutane, a direct precursor of the aminocyclopropanes.

3. Conclusion

The analogy between alkoxycarbene complexes of



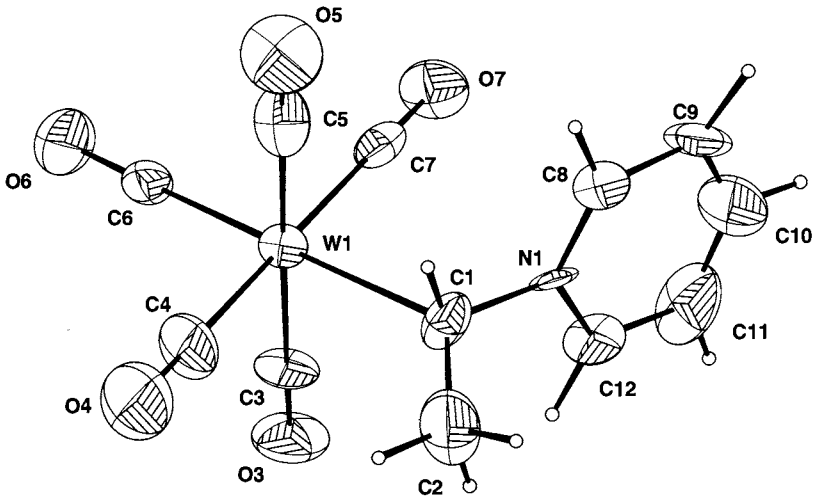


Fig. 3. CAMERON projection of complex **17** with the atomic numbering scheme.

tungsten and carbonyl compounds has been fruitfully applied to the reduction of carbene complexes with dihydropyridines: the reaction which is observed leads to a new class of alkylidene complexes of tungsten(0), which are stabilized as soon as they are formed by pyridine to give pyridinium ylide complexes.

In the case of the phenyl-substituted complex, an easy transfer of the carbene moiety was observed. This transfer is especially efficient with electron-rich olefins such as enol ethers and even more with enamines.

4. Experimental section

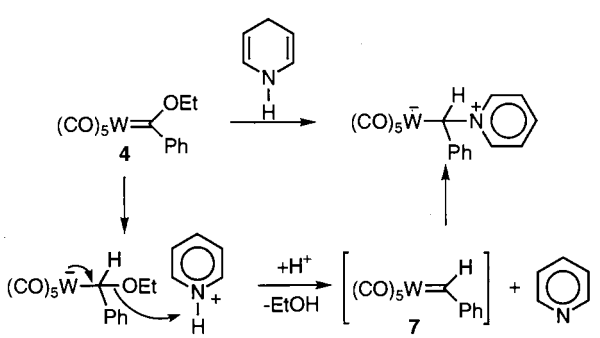
4.1. General methods

^{1}H and ^{13}C-NMR spectra were recorded on Bruker AC-200 or AMX-400 instruments. Mass spectra are *m/z*. Column chromatography was performed with Merck silica gel (70–230 mesh) using various ratios of dichloromethane/light petroleum ether and diethyl ether/light petroleum ether as eluent. All reagents were obtained from commercial suppliers and used as received. Reactions were performed under an argon atmosphere in carefully dried glassware. Solvents were dried by distillation from a drying agent: THF and Et_2O from Na/benzophenone; CH_2C1_2 from CaH_2 and P_2O_5. Enamines were prepared by published methods [13].

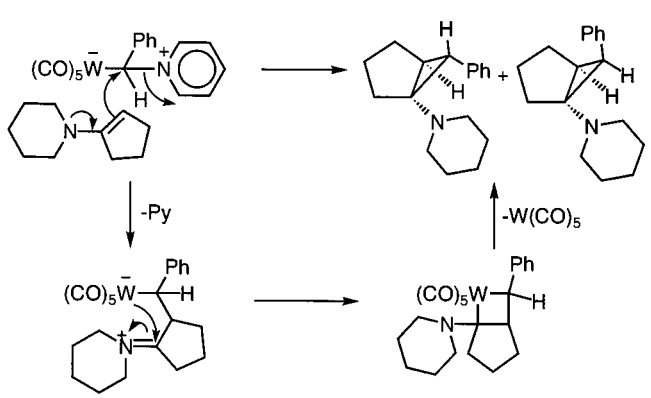
4.1.1. 7-Phenyl-2-oxa-bicyclo(4.1.0)heptane

N-Methyldihydropyridine (0.22 g, 2.3 mmol) was added to a solution of $(CO)_5W{=}C(OEt)Ph$ **4** (0.5 g, 1.14 mmol) in 25 ml of dichloromethane at −78°C. The solution turned instantly to dark red. After warming to 0°C, the solvent was evaporated under vacuum to give an oil which was washed with ice-cold hexane. The residue was then disolved in 15 ml of dichloromethane at −78°C and dihydropyrane (0.5 ml, 5.7 mmol) and acid trifluoroacetic (0.26 ml, 3.4 mmol) were added. The reaction mixture was warmed to r.t. slowly. Evaporation of solvent followed by silica gel chromatography gave with petroleum ether/dichloromethane (90/10) as eluent complex **14** and with petroleum ether/diethyl ether (90/10) **23** and **24** (0.07 g, 35%) as a melange of isomers (93/7).

Spectral data for **23** (major isomer): ^{1}H-NMR ($CDC1_3$) δ 7.43–7.20 (m, 5H, Ph), 3.88–3.85 (dd, 1H,



Scheme 3.



Scheme 4.

$J = 6,6$ Hz, H,), 3.51–3.45 (m, 1H, H_3), 3.33–3.21 (m, 1H, $H_{3'}$), 2.02–1.92 (m, 2H, H_5 and $H_{5'}$), 1.88–1.85 (m, 1H, H_7), 1.33–1.24 (m, 1H, H_6), 1.17 (m, 1H, H_4), 0.57 (m, 1H, $H_{4'}$). ^{13}C-NMR ($CDC1_3$) δ 131.8, 128.4, 126.2 and 121.6 (Ph), 64.8 (C_3), 53.1 (C_1), 23.3 (C_7), 22.8 (C_4), 17.5 (C_5), 13.7 (C_6). HRMS calc. (obsd) for $C_{12}H_{14}O^+$ 174.1044 (174.1044).

4.1.2. 1-[6-Phenyl-bicyclo(3.1.0)hex-l-yl]-piperidine

This was obtained from complex **25** (0.4 g, 0.88 mmol) and 1-cyclopent-1-enyl-piperidine (0.5 g, 3.3 mmol) in 10 ml of dichloromethane at r.t. for 1 h. Evaporation of the solvent followed by silica gel chromatography first gave with petroleum ether/dichloromethane (90/10) as eluent complex **14** as yellow cristals (0.2 g, 60%), and then with petroleum ether/diethyl ether (98/2) as eluent **26** (*trans*) as an oil (0.05 g, 24%) and (90/10) 27 (*cis*) as an oil (0.09 g, 46%).

Spectral data for **26**: ^{1}H-NMR ($CDC1_3$) δ 7.20–7.03 (m, 5H, Ph), 2.36 2.27 (m, 4H, N–CH2), 1.97–1.89 (m, 1H, H_3), 1.82 (d, 1H, $J = 4.6$ Hz, H6), 1.78–1.73 (m, 1H, H_5), 1.68–1.59 (m, 3H, H_4, H_5, and H_3), 1.53–1.51 (m, 1H, H_2), 1.31–1.18 (m, 7H, H_4, N–CH2–C$\underline{H}_2$, N–CH_2–CH_2–C$\underline{H}$2). ^{13}C-NMR ($CDC1_3$) δ 139.9, 128.2, 126.9, 124.6 (Ph), 60.8 (C_1), 50.7 (N–CH_2), 33.9 (C_2), 31.7 (C_6), 27.5 (C_5), 26.3 (N–CH_2–$\underline{C}H_2$), 24.8 (N–CH_2–CH_2–$\underline{C}H_2$), 23.3 (C_3), 21.5 (C_4).

Spectral data for **27**: ^{1}H-NMR ($CDC1_3$) δ 7.32–7.21 (m, 5H, Ph), 2.87–2.82 (m, 2H, CH$\underline{H}$–N–CH$\underline{H}$), 2.69–2.64 (m, 2H, C$\underline{H}$H–N–C$\underline{H}$H), 2.29–2.27 (d, 1H, $J = 9$ Hz, H_6), 2.06–1.98 (m, 1H, H_5), 1.95–1.86 (m, 1H, H_3), 1.70–1.57 (m, 7H, $H_{5'}$, N–CH_2–C$\underline{H}_2$, $H_{3'}$, H_2), 1.50–1.45 (m, 2H, N–CH_2–CH_2–C$\underline{H}_2$), 1.37–1.28 (m, 1H, H_4), 0.13–0.00 (m, 1H, H_4.). ^{13}C-NMR ($CDC1_3$) δ: 138.1, 128.5, 128.3, 125.9 (Ph), 59.2 (C_1), 512 (N–CH_2), 33 4 (C_6), 30.3 (C_2), 26.4 (N–CH_2–$\underline{C}H_2$), 25.3 (N–CH_2–CH_2–CH_2), 24.7 (C_3), 22.7 (C_4), 20.9 (C_5). HRMS calc. (obsd) for $C_{17}H_{23}N^+$ 241.1830 (241.1829).

4.1.3. 4-[7-Phenyl-bicyclo(3.1.0)hex-1-yl]-morpholine

This was obtained from complex **25** (0.4 g, 0.88 mmol) and 4-cyclopent-1-enyl-morpholine (0.5 g, 3.24 mmol) in 10 ml of dichloromethane at r.t. for 1 h. Evaporation of the solvent followed by silica gel chromatography first gave with petroleum ether/dichloromethane (90/10) as eluent complex **14** as yellow crystals (0.16 g, 50%), and then with petroleum ether/diethyl ether (98/2) as eluent **28** (*trans*) as an oil (0.1 g, 50%) and (90/10) **29** (*cis*) as an oil (0.06 g, 30%).

Spectral data for **28**: ^{1}H-NMR ($CDC1_3$) δ 7.28–7.13 (m, 5H, Ph), 3.55–3.43 (m, 4H, OCH_2), 2.52–2.47 (m, 4H, NCH_2), 2.09–2.01 (m, 1H, $H_{3'}$), 1.99–1.97 (d, 1H, $J = 4.3$ Hz, H_6), 1.91–1.84 (m, 1H, H_5), 1.82–1.74 (m, 3H, H_4, $H_{5'}$ and H_3), 1.70–1.68 (m, 1H, H_2), 1.44–1.38 (m, 1H, $H_{4'}$). ^{13}C-NMR ($CDC1_3$) δ 128.4, 127.1, 125.0 and 121.4 (Ph), 67.3 (OCH_2), 60.1 (C_1), 49.6 (NCH_2), 33.1 (C_6), 31.6 (C_2), 29 3 (C_5), 28.5 (C_4), 23.5 (C_3).

Spectral data for **29**: ^{1}H-NMR ($CDC1_3$) δ 7.32–7.21 (m, 5H, Ph), 3.75–3.73 (m, 4H, OCH_2), 2.91–2.86 (m, 2H, C$\underline{H}$H–N–C$\underline{H}$H), 2.75–2.70 (m, 2H, CH$\underline{H}$–N–CH$\underline{H}$), 2.29–2.27 (d, 1H, $J = 8.7$ Hz, H_6), 2.06–1.97 (m, 1H, H_3), 1.96–1.86 (m, 1H, H_5), 1.71–1.69 (m, 1H, H_2), 1.68 1.60 (m, 2H, $H_{3'}$ and $H_{5'}$), 1.40–1.32 (m, 1H, H_4), 0.15–0.02 (m, 1H, $H_{4'}$). ^{13}C-NMR ($CDCl_3$) δ 137.6, 128.9, 128.3 and 126.Ph), 67.5 (OCH_2), 59.1 (C_1), 50.1 (NCH_2), 33.3 (C_6), 29.9 (C_2), 25.3 (C_5), 22.6 (C_4), 21.2 (C_3). HRMS calc. (obsd) for $C_{16}H_{21}NO^+$ 243.1623 (243.1623).

4.1.4. 4-[7-Phenyl-bicyclo(4.1.0)hept-1-yl]-morpholine

This was obtained from complex **25** (0.5 g, 1 mmol) and 4-cyclohex-1-enyl-morpholine (0.85 g, 5 mmol) in 10 ml of dichloromethane at r.t. for 1 h. Evaporation of the solvent followed by silica gel chromatography first gave with petroleum ether/dichloromethane (90/10) as eluent complex **14** as yellow crystals (0.1 g, 30%), and then with petroleum ether/diethyl ether (98/2) as eluent **30** (*trans*) as an oil (0.075 g, 30%) and (90/10) 31 (*cis*) as an oil (0.05 g, 18%).

Spectral data for **30**: ^{1}H-NMR ($CDC1_3$) δ 7.34–7.12 (m, 5H, Ph), 3.51–3.48 (m, 4H, OCH_2), 2.55–2.51 (m, 4H, NCH_2), 2.16–2.09 (m, 1H, $H_{6'}$), 1.99–1.90 (m, 1H, H_4), 1.80–1.71 (m, 2H, H_7 and $H_{4'}$), 1.67–1.60 (m, 1H, H_6), 1.49–1.21 (m, 5H, H_2, H_3, $H_{3'}$, H_5 and $H_{5'}$). ^{13}C-NMR ($CDC1_3$) δ 129.9, 128.0, 127.2 and 125.1 (Ph), 67.3 (OCH_2), 50.3 (C_1), 48.4 (NCH_2), 36.5 (C_7), 28.2 (C_2), 24.1 (C_6), 22.5 (C_5), 22.4 (C_4), 21.3 (C_3).

Spectral data for **31**: ^{1}H-NMR ($CDC1_3$) δ 7.22–7.10 (m, 5H, Ph), 3.63–3.60 (m, 4H, OCH_2), 2.69–2.66 (m, 4H, NCH_2), 1.99–1.92 (m, 2H, H_6 and H_7), 1.85–1.75 (m, 2H, H_3 and $H_{3'}$), 1.38–1.26 (m, 2H, H_2 and $H_{6'}$), 1.06–1.01 (m, 1H, H_4), 0.94–0.82 (m, 2H, H_5 and $H_{5'}$), 0.43–0.33 (m, 1H, $H_{4'}$). ^{13}C-NMR ($CDC1_3$) δ 138.4, 135.6, 128.9 and 126.6 (Ph), 68.0 (OCH_2), 49.1 (NCH_2), 46.9 (C_1), 32.9 (C_7), 24.1 (C_2), 23.1 (C_5), 22.3 (C_4), 21.8 (C_3), 18.8 (C_6). HRMS calc. (obsd) for $C_{17}H_{23}NO^+$ 257.1780 (257.1780).

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