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Synthesis and Multi-Nuclear NMR study on phosphorus ylide containing oxophilic group 4 metal complexes

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The utility of metalated phosphorus ylides in synthetic chemistry has been well documented. The decahalodimetallate $[M_2Cl_{10}]^{2-}$ anions of zirconium(IV) and hafnium(IV) within the second and third row of the transition d elements have been prepared as their triphenylmethyl derivatives $(Ph_3C)_2[M_2Cl_{10}]$. In view of these observations we have initiated an investigation of the chemistry of $TiCl_4$, $ZrCl_4$ and $HfCl_4$ with phosphorus ylides as a ligand. Dimethyl sulfoxide is an ambidentate ligand, usually coordinating via the oxygen atom, but to a number of soft electron-pair acceptors, it is also coordinating via the sulfur atom. The structure of the dimethyl sulfoxide solvated zirconium(IV) ion has been reported in solid octakis(dimethyl sulfoxide)zirconium(IV) chloride dimethyl sulfoxide. In this work we study reactions between oxophilic group 4 metal chlorides, α-keto ylide, ($Ph_3PCHCOPhCH_3$), in THF, led to the formation of $(Ph_3PCH_2COPhCH_3)_2$ $[M_2Cl_{10}]$(M= Zr(1), Hf (2)and Ti(3)). After adding dimethyl sulfoxide to these compounds in room temperature crystalline solid $M(DMSO)_8.4Cl.mH_2O.DMSO$ (M=Ti, Zr and Hf), together with $Ph_3PCH_2COPhCH_3$ in mother liquid was formed.The aims of our present work are to describe the preparation, spectroscopic characterization of complexes 1, 2, 3.

Keywords: Phosphorus ylide, Zirconium(IV), Titanium(IV), Hafnium(IV).

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One-Pot Synthesis of β -Acetamido Ketones and Esters Using Bulk and Activated Carbon-Supported Tungstophosphoric Acid as an Inorganic Green Catalyst

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Heteropoly acids are solid inorganic polyacids, with great applications in many areas and can be used either directly as a bulk material or in supported form. The supported form is preferable because of its higher surface area and better accessibility of reactants to the active sites. Based on our previous studies on the use of heteropoly acid catalysts, herein we wish to report a rapid and efficient catalytic procedure for the one-pot synthesis of β-acetamido ketones and esters using tungstophosphoric acid (PW) as bulk and supported on activated carbon. After impregnation of activated carbons with PW, surface area, pore volume, and average pore diameters were decreased. N_2 isotherms and pore volume distribution are investigated. Supported PW acid yielded much higher conversion than bulk form. In conclusion, an efficient method for the synthesis of β -acetamido carbonyl compounds is developed in the presence of a new synthesized activated carbon-supported Keggin catalyst. Simple experimental procedure, high yield and selectivity, makes this method useful addition to the present methodologies.

Keywords: Tungstophosphoric acid**,** Heteropoly acid, Catalyst, β-acetamido ketone