Inorgano-Grignard Reagents. Preparation, Reactions, and *X*-Ray Crystal Structure of Bis-(η-cyclopentadienyl)hydridomolybdenum[bromo-(bistetrahydrofuran)magnesium]

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Summary The crystal and molecular structure determination of $[(\eta\text{-}C_5H_5)_2MoHMg(C_4H_8O)_2Br]$ reveals the presence of an Mo–Mg bond, length 2·732 Å; the title compound can act as both a nucleophile and a base.

RECENTLY interest has developed in compounds containing covalent bonds between a transition metal and magnesium as possible intermediates in the activation of Grignard reagents by transition metal compounds.[1] However, few compounds containing covalent transition metal–magnesium bonds have been reported.[2,3]

The compound $[(\eta\text{-}C_5H_5)_2MoHMg(C_3H_7)(\mu\text{-}Br_2)Mg(Et_2O)]_2$ (I) has been previously described.[3] Dissolving the yellow crystalline compound (I) in THF (tetrahydrofuran) gave a red solution which, on concentration, yielded an orange crystalline compound (II) (80%). The crystal structure of the product (II) has been determined. *Crystal data*: $(\eta\text{-}C_5H_5)_2Mo(H)Mg(OC_4H_8)_2Br$, $M = 475{\cdot}1$, triclinic, $a = 11{\cdot}708(5)$, $b = 11{\cdot}596(5)$, $c = 7{\cdot}750(3)$ Å, $\alpha = 86{\cdot}33(2)$, $\beta = 8292(2)$, $\gamma = 73{\cdot}26(3)°$; space group $P\bar{1}$ (C_i^1, No. 2), from intensity statistics and structure analysis, 4-circle diffractometer data, monochromated Mo-K_α radiation ($\mu = 49{\cdot}3$ cm^{-1}), 1334 independent reflections, $I > 3\sigma(I)$. The structure was determined by the heavy atom method and refined by full matrix least squares to give a conventional R of 6·6%. The hydrido hydrogen and the hydrogen atoms of the tetrahydrofuran could not be found in the difference syntheses. The structure is shown in the Figure. The Mo–Mg bond length is 2·732 Å, the same as that reported previously[4] for an Mo–Mg bond in which the Mo acts as a one-electron donor. The i.r. spectrum of (II) shows a band at 1850 cm^{-1} which may be assigned to ν (Mo–H).

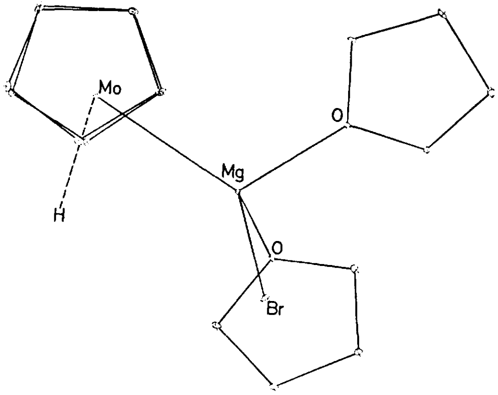
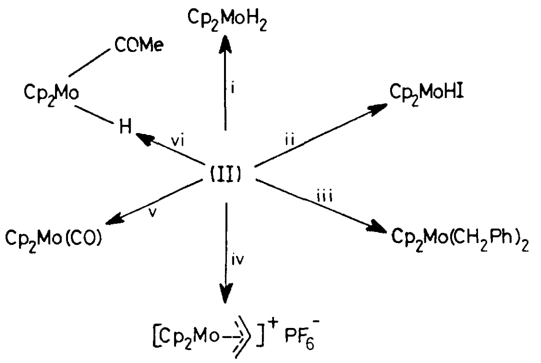
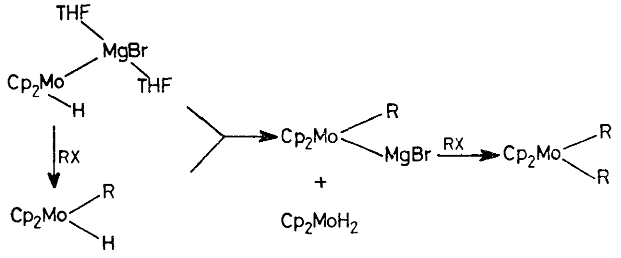


FIGURE. Structure of $[(\eta\text{-}C_5H_5)MoHMg(THF)_2Br]$ (II). The postulated position of the hydrido hydrogen is shown in broken lines and lies in the plane of the paper.



SCHEME 1. Some reactions of $[Cp_2MoHMg(THF)_2Br]$ in THF at 20 °C. Cp = $\eta\text{-}C_5H_5$. (i) H_2O or anhydrous acetone (100% yield); (ii) MeI (20%); (iii) $PhCH_2X$ (X = Cl or Br) (43%); (iv) $CH_2{:}CHCH_2Br$ then aqueous NH_4PF_6 (24%); (v) CO_2 (45%); (vi) MeCOCl (75%).

The Mo–Mg compound (II) is soluble in THF from which it is recrystallisable. It is very sensitive to air and is instantly hydrolysed. Scheme 1 shows the reactions of (II). The compound is highly reactive behaving either as a nucleophile or as a base.



SCHEME 2. Possible mechanism for the reaction of $[Cp_2MoHMg(THF)_2Br]$ with RX.

A mechanism accounting for the variety of products obtained from the reactions of (II) is shown in Scheme 2. A similar mechanism may also be proposed to explain the previously reported reactions of (I).

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