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THE PREPARATION OF (3-3-(PH3P)2-3-H-4-(POLYSTYRYLMETHYL)-3-1-2---ETC(U)

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TECHNICAL REPORT NO. 97

The Preparation of $[3,3-(\text{Ph}_3\text{P})_2-3\text{-H-4-(polystyrylmethyl)-3,1,2-}\text{RhC}_2\text{B}_9\text{H}_{10}]$; A Polymer-Bound Metallocarborane Catalyst.

By

B. A. / Sosinsky, W. C. / Kalb, R. A. / Grey, V. A. / Uski and
M. F. / Hawthorne

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[Contribution No. 3825 from the Department of Chemistry,
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The Preparation of $[3,3-(\text{Ph}_3\text{P})_2-3\text{-H-4-(polystyrylmethyl)-3,1,2-}$
 $\text{RhC}_2\text{B}_9\text{H}_{10}]$; A Polymer-Bound Metallocarborane Catalyst

By

B. A. Sosinsky, W. C. Kalb, R. A. Grey, V. A. Uski, and
M. F. Hawthorne*

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ABSTRACT

The compound $[3,3-(\text{Ph}_3\text{P})_2-3\text{-H-4-(polystyrylmethyl)-3,1,2-RhC}_2\text{B}_9\text{H}_{10}](\text{I})$ was synthesized from $[(\text{Ph}_3\text{P})_3\text{RhCl}]$ and $[9\text{-(polystyrylmethyl)-7,8-C}_2\text{B}_9\text{H}_{11}]^-\text{M}^+$ (II; M = Na or K). Characterization of (I) includes infrared, visible, and X-ray photoelectron spectroscopy and elemental analysis. The polymer-bound carborane (II) was isolated from the reaction of $(7,8\text{-C}_2\text{B}_9\text{H}_{11})^{2-}$ with Merrifield's peptide resin. Complex (I) is an active catalyst for the hydrogenation of olefins, with a rate roughly comparable to its homogeneous analogue $[3,3-(\text{Ph}_3\text{P})_2-3\text{-H-3,1,2-RhC}_2\text{B}_9\text{H}_{11}](\text{III})$.

Sir:

Considerable interest has recently developed in the attachment of homogeneous transition metal catalysts to insoluble supports in order to aid in product isolation and in catalyst retrieval.¹⁻⁵ This concept of supporting active catalytic molecules was initially developed^{6,7} by biochemists who utilized bound enzymes as their catalysts. Inorganic approaches to this area have centered on anchoring transition metal catalysts to both polar supports^{8,9} such as silica and to non-polar supports¹⁰⁻¹⁴ such as functionalized organic polymers either directly or via bound ligands.^{10-13, 15-20}

We wish to report the synthesis and characterization of [3,3-(Ph₃P)₂-3-H-4-(polystyrylmethyl)-3,1,2-RhC₂B₉H₁₀] (I), the first supported metallocarborane catalyst (polymer-bound by a carbon-boron bond), and an analogue of the previously reported²¹ homogeneous hydrogenation catalyst [3,3-(Ph₃P)₂-3-H-3,1,2-RhC₂B₉H₁₁] (III). The preparation of polymer catalyst (I) outlined in Scheme 1 involved the intermediate preparation of M⁺(9-(polystyrylmethyl)-7,8-C₂B₉H₁₁)⁻ (II; M = Na, K) by reaction of dicarbollide dianion^{22,23} with chloromethylated polystyrene polymer²⁴ (Merrifield's peptide resin) in the presence of dibenzo-18-crown-6. The crown ether was used as a catalyst to aid in the transfer of dianion into the lipophilic polymer. Reaction of the carborane substituted polymer (II) with [(Ph₃P)₃RhCl] yielded the rhodacarborane polymer-bound catalyst (I).

Supported metallocarborane catalysts exhibit a distinctive advantage not found in previously explored polymer-bound catalysts. Systems in which a triaryl phosphine ligand is attached to the polymer and serves as the metal-polymer linkage,^{12,13,17,25} requires reversible phosphine dissociation in the catalysis mechanism and may be susceptible to eventual loss of metal content. The strong metal-carborane bond should be retained and the catalytic metal center rendered immobile.

Scheme 1

The alkylation of $(7,8-C_2B_9H_{11})^{2-}$ with alkyl halides had been found previously to occur at the boron atom adjacent to the carbon in the open pentagonal face (positions 9 and 11) and to a lesser extent at the boron atom in between and below the two carbon atoms (position 3).²⁶ Heating the 9-isomer (R = Me, and Et) at 65° for short periods completely isomerizes it to the 3-isomer.²⁶ Alkylation of $(7,8-C_2B_9H_{11})^{2-}$ with benzyl chloride under identical reaction conditions with which the polymer is made yielded a 4:1 mixture of the 9-benzyl and 3-benzyl substituted cage. Based on these findings, we believe that the carborane in (I) and (II) is predominantly bound to the polymeric benzyl groups at the 9-position, and to a somewhat lesser extent at the 3-position.

The infrared spectrum (KBr) of $(9-(\text{polystyrylmethyl})-7,8-C_2B_9H_{11})^-$ (II) exhibits a strong ν_{BH} stretch centered at 2058 cm^{-1} . Compound (II) is a white solid which is stable in air for extended periods. However, (II) was handled under an inert atmosphere whenever possible to prevent contamination with water as $K(C_2B_9H_{12})$ is quite hygroscopic.²³ Elemental analysis of (II) demonstrated the incorporation of 6.72% boron. (C = 79.24, H = 8.11, K = 1.30, Na = 0.69, and Cl = 0.00%). These data suggests quantitative cage incorporation into the chloromethylated polystyrene matrix.

The infrared spectrum (KBr pellet) of $[3,3-(Ph_3P)_2-3-H-4-(\text{polystyrylmethyl})-3,1,2-RhC_2B_9H_{10}]$ (I) contains a B-H stretching absorption at 2560 cm^{-1} and a weak ν_{RhH} stretch at 2080 cm^{-1} . No other characteristic bands were observed due to the low concentration of embedded metal complex and also to the obscuration of several spectral regions by the polymer-backbone. Complex (I) is a bright yellow air-

stable material with a color identical to that of its homogeneous analogue [3,3-(Ph₃P)₂-3-H-3,1,2-Rh(C₂B₉H₁₁)] (III). Elemental analyses of several preparations of the polymer-bound complex (I) were determined and are typified by the following figures: C = 76.76, H = 6.88, B = 4.90, Rh = 4.42, P = 2.33, and Cl = 0.62%.²⁷ This analysis established ratios of cage/Cl = 2.9,²⁷ Rh/cage = 0.85; and P/Rh = 1.8.²⁸ The residual percentage of the compound not found in the analysis was 4.09%, presumably, due to the presence of oxygen along with some sodium or potassium.²⁷ Other determinations of (I) established cage/Cl ratios of 0.37-5.2,²⁷ Rh/cage ratios of 0.27-0.85, Rh/P ratios of 1.75-2.7, and residual percentage of undetermined elements of 2.9-5.4%.^{27,28}

The diffuse visible reflectance spectra of both [3,3-(Ph₃P)₂-3-H-4-(polystyrylmethyl)-3,1,2-RhC₂B₉H₁₀] (I) and [3,3-(Ph₃P)₂-3-H-3,1,2-RhC₂B₉H₁₁] (III) were measured (relative to MgO). Both spectra are identical in shape and exhibited broad absorption with a maxima at 444.5 nm. Measurement of the visible and UV spectrum of (III) (C₆H₆) reveals two absorptions at: 415 nm (ε = 930 l/Mcm), and 325 nm (ε = 9,500 l/Mcm), the latter of which appears as a shoulder on an intense charge-transfer band. The two bands arise²⁹ from transitions from ¹A₁ ground state to ¹T₁ and ¹T₂ upper states. The extinction coefficient of the absorption at 325 nm is increased by intensity stealing from the charge transfer band.

The X-ray photoelectron spectra (ESCA) of both (I) and (III) were measured for the phosphorus 2p_{3/2}, boron 1s, and rhodium 3d_{5/2} and 3d_{3/2} levels. The phosphorus and boron signals in (III) and (I) are broad but of nearly the same shape and have their maxima at: boron at 188.7 and 188.9, and phosphorus at 131.4 and 131.4 eV, respectively. The rhodium levels for (III) and (I) were found at: 3d_{3/2} at 312.4 and 312.5, and 3d_{5/2} at 307.8 and 307.9, respectively. This data is shown graphically in Figure 1.

Figure 1

In general, valence electron binding energy levels as measured by the X-ray photoelectron spectroscopy do not change much with an oxidation state change.^{31,32} Rhodium foil exhibits its $3d_{3/2}$ and $3d_{5/2}$ levels at 311.8 and 307.0 eV, respectively. However, small differences in the core 3d levels are significant³¹ and the close agreement between the maxima for the rhodium levels in (I) and (III) suggest that the majority of the rhodium atoms incorporated into the polymer beads (at least at the surface) have an identical chemical environment to the rhodium atoms in $[3,3-(\text{Ph}_3\text{P})_2-3\text{-H-3,1,2-RhC}_2\text{B}_9\text{H}_{11}]$. These measurements are only indicative of the surface environments. However, with a highly porous polymer considerably more surface area may be exposed than with a normal crystal. Measurement of the UV photoelectron spectra (UPS) may yield a more definitive comparison as UPS measures valence levels and is therefore more sensitive to oxidation state changes.³²

The beads (I) in an amount equivalent to a 10^{-4} M solution of (III) in benzene isomerize 1-octene (0.3 M) within 24 hours at 40° to a mixture of 1-octene (14%), trans-2-octene (65%), and cis-2-octene (20%). Reaction of (I) with deuterium gas at 300 psi and 45° for 2 days yielded tan beads with a considerable amount of cage-deuterium incorporation as shown in the infrared.³³

The compound $[3,3-(\text{Ph}_3\text{P})_2-3\text{-H-4-(polystyrylmethyl)-3,1,2-RhC}_2\text{B}_9\text{H}_{10}]$ (I) was found to be an efficient catalyst for the reduction of (blocked) alkenes to alkanes as has been demonstrated^{21,34} for its homogeneous analogue $[3,3-(\text{Ph}_3\text{P})_2-3\text{-H-3,1,2-RhC}_2\text{B}_9\text{H}_{11}]$ (III). Kinetic runs of (I) were made in a flask containing a vortex plug with a hole through the center so that polymer beads would remain suspended in the solution (and not be deposited on the walls of the flask above the solution level), and with a stir bar rotating at maximum speed. The rate of reduction may be diffusion controlled and a more efficient stirring system might increase the rate somewhat. Hydrogenation of

3,3-dimethyl-1-butene (0.39 M) using the bead catalyst (I) proceeded at a rate of 2.45×10^{-3} ml H_2 (1 atm)/min x mg(I) (or 9.52×10^{-5} mol/min x g(I)) at 40.0° in ortho-dichlorobenzene solution and ethyl acrylate reduction in ortho-dichlorobenzene with (I) (at 0.67 M ethyl acrylate) occurred at an initial rate of 3.79×10^{-3} ml H_2 (1 atm)/min x mg(I) (or 4.7×10^{-5} mol/min x g(I)).

Under a hydrogen atmosphere and in the presence of alkene solutions bead catalyst (I) is yellow. Upon consumption of the olefin the beads change to a brown color. The homogeneous catalyst (III) shows no such color change in the absence of oxygen. Elaborate measures to exclude oxygen failed to prevent the color change in hydrogenation reactions of (I). However, with 3,3-dimethyl-1-butene the supported catalyst (I) has been observed to go over 250 turnovers (per Rh atom) without changing color, and to continue hydrogenating 3,3-dimethyl-1-butene roughly 20% slower for an additional 280 turnovers (the length of the experiment) after becoming brown. No color change has been observed over a period of several weeks for (I) in air either as a solid or in solution. Attempts to induce the color change by reaction of (I) with air either in the presence of 3,3-dimethyl-1-butene or in its absence left the beads an unchanged yellow color after several days. The reason for the color change after hydrogenation is not known at this time.

In addition to the method we have demonstrated for the attachment of carborane cages to polymers, a variety of new routes and extensions to polymeric metallocarboranes may easily be envisaged. Considerable derivative chemistry of carboranes exists which would allow not only the attachment of the polymer to cages in various locations, but also homo- and co-polymerizations of monomers already containing boron cages.^{35,36}

Acknowledgements

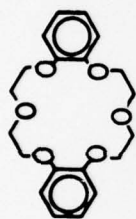
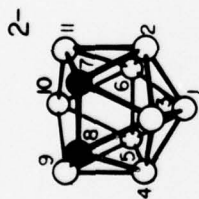
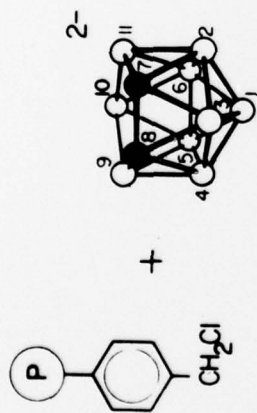
We wish to thank the Office of Naval Research for the support of this work, and Matthey-Bishop, Inc. for a generous loan of rhodium trichloride.

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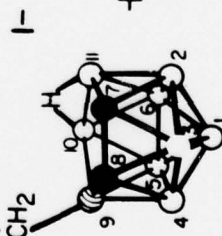
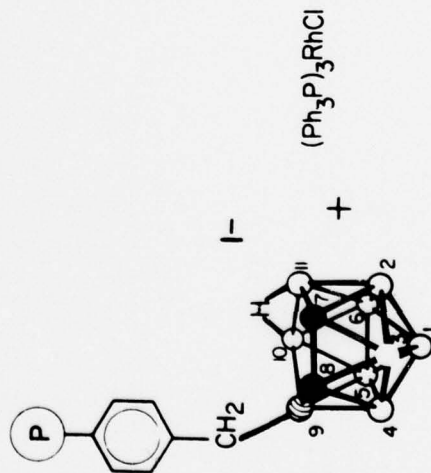
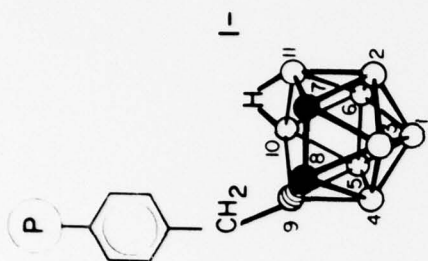
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Scheme 1. The synthesis of $[3,3-(\text{Ph}_3\text{P})_2-3\text{-H-4-(polystyrylmethyl)-}$
 $3,1,2\text{-RhC}_2\text{B}_9\text{H}_{10}]$.

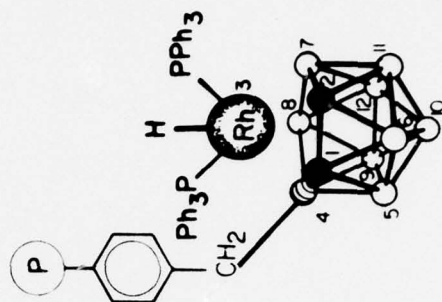


THF/66°C
24 HOURS



(Ph₃P)₃RhCl

C₂H₅OH/78°C
12 HOURS



● = CH
○ = BH
⊙ = B

Figure 1. X-ray photoelectron spectra (ESCA) of $[3,3-(\text{Ph}_3\text{P})_2-3\text{-H-}3,1,2\text{-RhC}_2\text{B}_9\text{H}_{11}]$ (III) and $[3,3-(\text{Ph}_3\text{P})_2-3\text{-H-}4\text{-(polystyrylmethyl)-}3,1,2\text{-RhC}_2\text{B}_9\text{H}_{10}]$ (I).

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