Experimental Section

General Considerations: All reactions were performed in the fume hood unless otherwise specified. ATR-IR was collected on a Thermo Scientific FTIR spectrometer

$CuSO_4\cdot5H_2O$ (0.53 g, 3.32 mmol) was ground into a fine powder and added to an Erlenmeyer flask containing triphenylphosphine (2.49 g, 9.49 mmol), 200-proof ethanol (40 mL), and a magnetic stir bar. The mixture was stirred with mild heating (~40 °C) until dissolution was complete. Any insoluble blue residue was removed by decantation. Still stirring, 0.50 g $NaBH_4$ was added in small batches to induce precipitation; addition continued until gas evolution ceased. The precipitate was collected by filtration, dissolved in chloroform (10 mL), and stirred until no further dissolution occurred. Any remaining insoluble inorganic salts were removed by filtration. The chloroform filtrate was warmed to ~70 °C, and ethanol (7 mL) was added dropwise to induce crystallization. The solution was allowed to cool slowly to room temperature and then placed in an ice bath to complete precipitation. The resulting white, crystalline solid was collected by filtration, dried, and weighed to obtain yield (0.97 g, 51.32%, 1.61 mmol). The product was characterized by ATR-IR. IR (cm^{-1}): 3052.28, 2399.01, 1989.70, 1478.65, 1432.85, 1140.69, 1095.37, 972.91, 741.01, 691.36, 494.17, 485.49

Table 1: IR vibrations commonly observed for various M-BH_4 structures

Structure	***Frequency (cm^{-1})***	***Type of motion***	***Symmetry Label***	***Notes***
Monodentate	2300-2450	B–H_{term} (stretch)	A_1, E	Strong, likely a doublet
	~2000	B–H_{bridg} (stretch)	A_1	Strong
	1000-1150	BH_3 (deformation)	A_1, E	Strong band with weaker shoulder at slightly higher frequency
Bidentate	2400-2600	B–H_{term} (stretch)	A_1, B_1	Strong doublet, 40–80 cm^{-1} splitting
	1950-2150	B–H_{bridg} (stretch)	A_1, B_2	Strong band with possible shoulder
	1400-1500	Bridge (stretch)	A_1	Strong
	1100-1200	BH_2 (deformation)	B_2	Strong
Tridentate	2450-2600	B–H_{term} (stretch)	A_1	Strong singlet
	2100-2200	B–H_{bridg} (stretch)	A_1, E	Doublet, 50-80 cm^{-1} splitting
	1150-1250	Bridge (deformation)	E	Strong

IR Spectrum

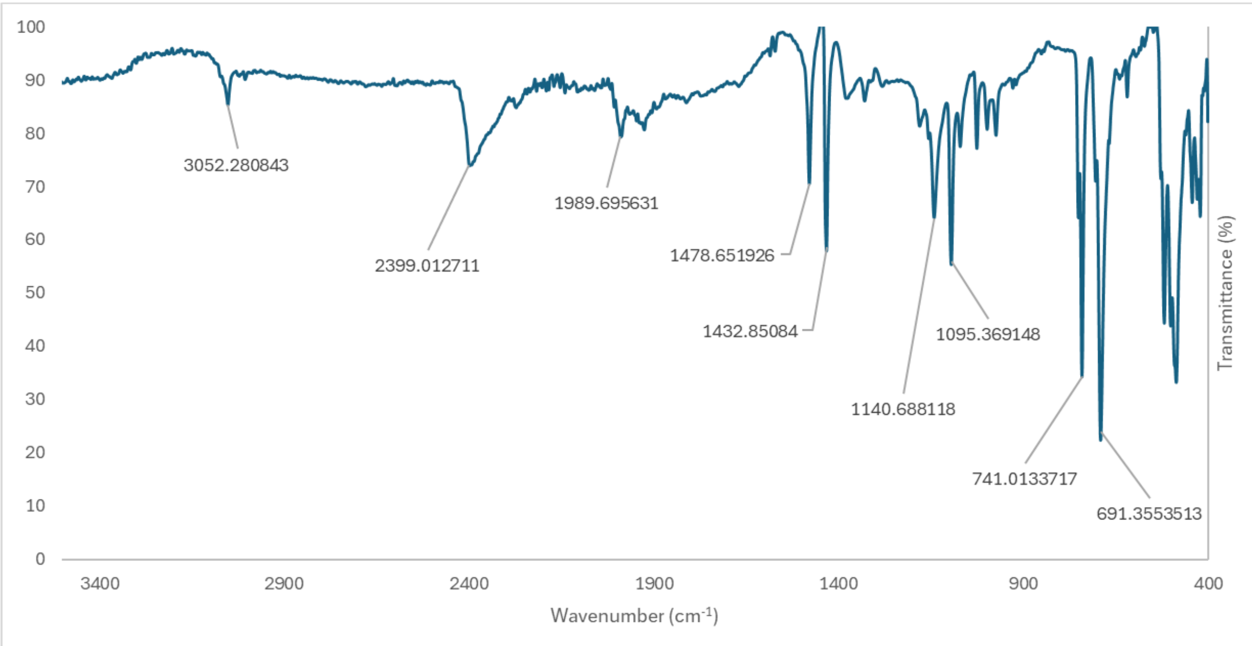

Figure 1:ATR-IR spectrum of synthesized $Cu(PPh_3)_2BH_4$ product. Peaks at 3052 cm^{-1}: aromatic C-H stretch from unreacted PPh_3. 2399 cm^{-1}: terminal B-H stretch of product. 1990 cm^{-1}: bridged B-H stretch of product.1477 cm^{-1} & 1433 cm^{-1}: doublet of bridged B-H stretch of product OR doublet of aromatic C=C stretch/in-plane C-H bending from unreacted PPh_3. 1141 cm^{-1} & 1095 cm^{-1}: doublet of BH_X deformation of product. 741 cm^{-1} & 691 cm^{-1}: doublet of out-of-plane C-H bending of monosubstituted benzene ring from unreacted PPh_3.

The data is inconclusive. It could be bidentate since that is the only structure with any signal at 1400-1500 cm^{-1}, and there are also signals present between 1100-1200 cm^{-1} that correspond with Table 1. The notes don't specify the shape of the peak – only that it be strong – but it's worth noting that both signals are doublets, which are not explicitly mentioned in Table 1. The signal at 1990 cm^{-1} is very weak, which contradicts the expected strong band between 1950-2150 cm^{-1}. A strong doublet between 2400-2600 cm^{-1} is also expected, and while there is a signal present at 2399 cm^{-1}, it is a broad, weak peak. The case could also be made for a monodentate structure as the expected strong band with a weaker shoulder is present between 1000-1150 cm^{-1}. There *are* signals around 2000 cm^{-1} and between 2300-2450 cm^{-1}, but again, these signals are very weak and there is no doublet between 2300-2450 cm^{-1}. There could also be unreacted PPh_3 ligands present: PPh_3 IR spectra show strong doublets at 697 cm^{-1} & 742 cm^{-1} and 1435 cm^{-1} & 1475 cm^{-1}, and a weak signal around 3000 cm^{-1}. This could explain the presence of unexpected peaks in a monodentate spectrum. Due to time constraints, ATR-IR was performed instead of solution cell IR or with KBr pellets. Since the compound is a white, crystalline substance, it is possible that the crystal structure resulted in variable surface contact and air gaps; this can lead to scattered IR radiation and non-uniform interactions, resulting in weak peaks and distorted intensities. If these discrepancies are truly due to messy optical interaction (meaning we ignore the intensities), the presence of signals at 1095 cm^{-1}& 1141 cm^{-1}, 1433 cm^{-1} &1479 cm^{-1}, 1990 cm^{-1}, and 2399 cm^{-1} corresponds to the expected signals of a bidentate structure. The broadness of the peak at 2399 cm^{-1} could be a result of the expected doublet signal being obscured, but I am unsure whether messy optics can change a peak structure so drastically. The same reasoning also applies to a possible monodentate spectrum with unreacted PPh_3 ligands: the observed signals match well with the expected signals, but the correct peak intensities and diagnostic terminal B-H doublet are not present.