

The  $^{13}\text{C}$  NMR spectrum of a sample of  $\text{Ta}_2\text{Cp}'_2\text{Cl}_4\text{H}(^{13}\text{CHO})$  in bromobenzene to which two equivalents of HCl had been added showed (besides a peak due to some unreacted starting material) a methoxide carbon peak at 66.84 ppm ( $J_{\text{CH}} = 146$  Hz, major product) and another at 62.85 ppm ( $J_{\text{CH}} = 146$  Hz, minor product).

If only one equivalent of HCl is used, half the starting material remains.

#### Reaction of $\text{Ta}_2\text{Cp}'_2\text{Cl}_4\text{H}(\text{CHO})$ with $\text{AlMe}_3$

$\text{AlMe}_3$  (0.14 g, 2 mmol) was added to a toluene solution of  $\text{Ta}_2\text{Cp}'_2\text{Cl}_4\text{H}(\text{CHO})$  (0.42 g, 0.5 mmol) at  $-30^\circ$ . The color changed from red to clear yellow. Solvent was removed in vacuo and the yellow oil was dissolved in  $\text{C}_6\text{D}_6$ . An  $^1\text{H}$  NMR spectrum showed a hydride resonance at 8.3 ppm and a formyl proton resonance at 6.6 ppm (d,  $J = 4$  Hz).

In a separate experiment excess  $\text{AlMe}_3$  was added to a solution of  $\text{Ta}_2\text{Cp}'_2\text{Cl}_4\text{H}(^{13}\text{CHO})$  in  $\text{C}_6\text{D}_6$ . The  $^{13}\text{C}$  NMR spectrum showed formyl carbon resonances at 227.47 ppm (d,  $J = 156$  Hz) and 214.36 ppm (dd,  $J = 152.6, 9.1$  Hz).

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