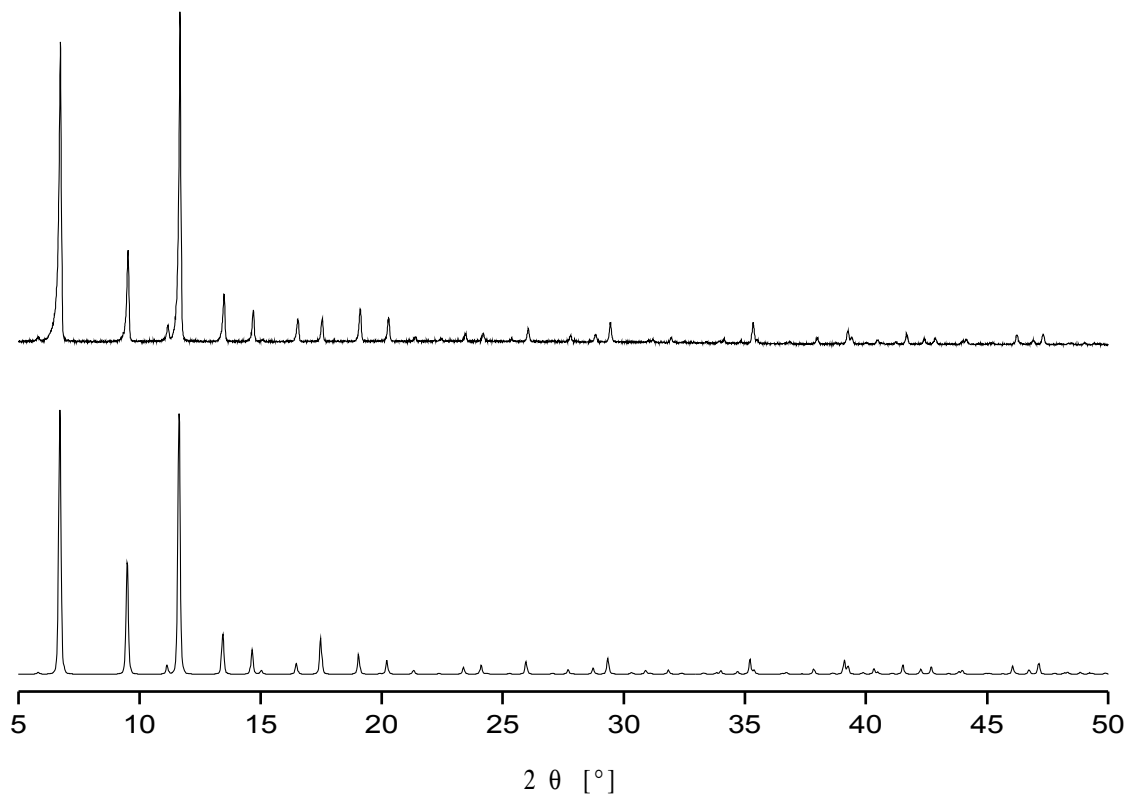
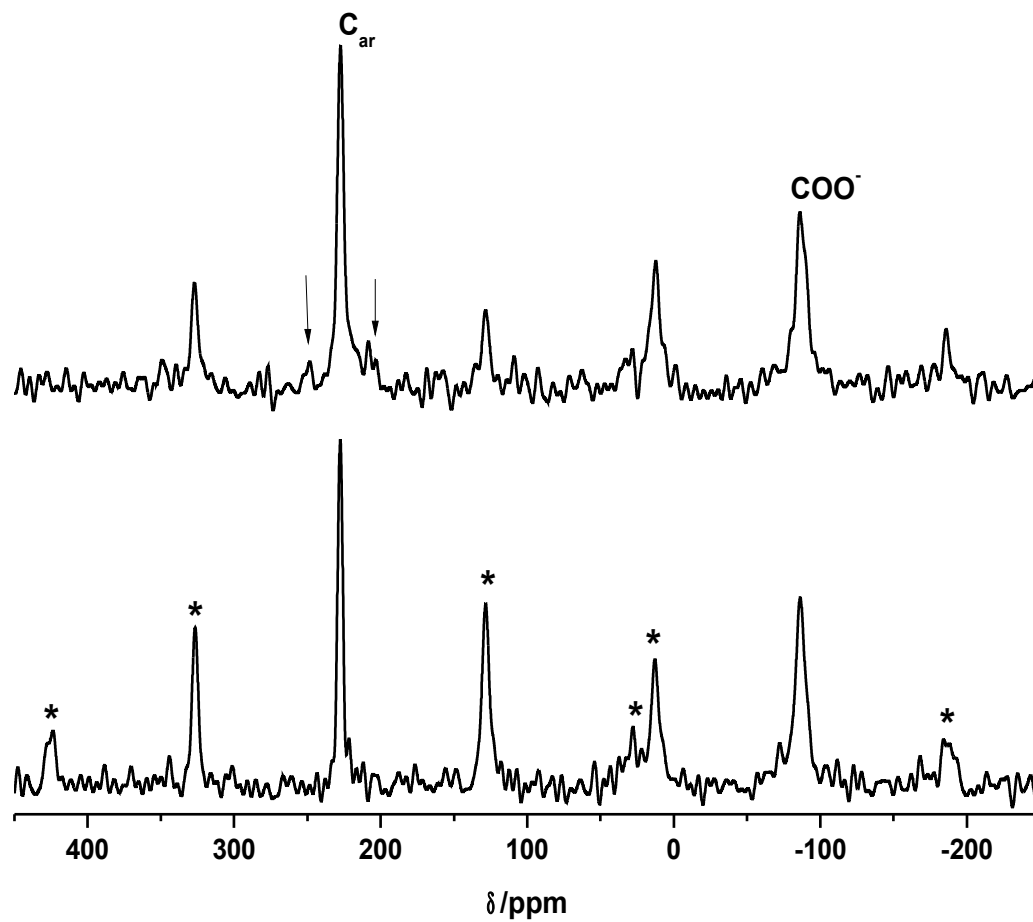


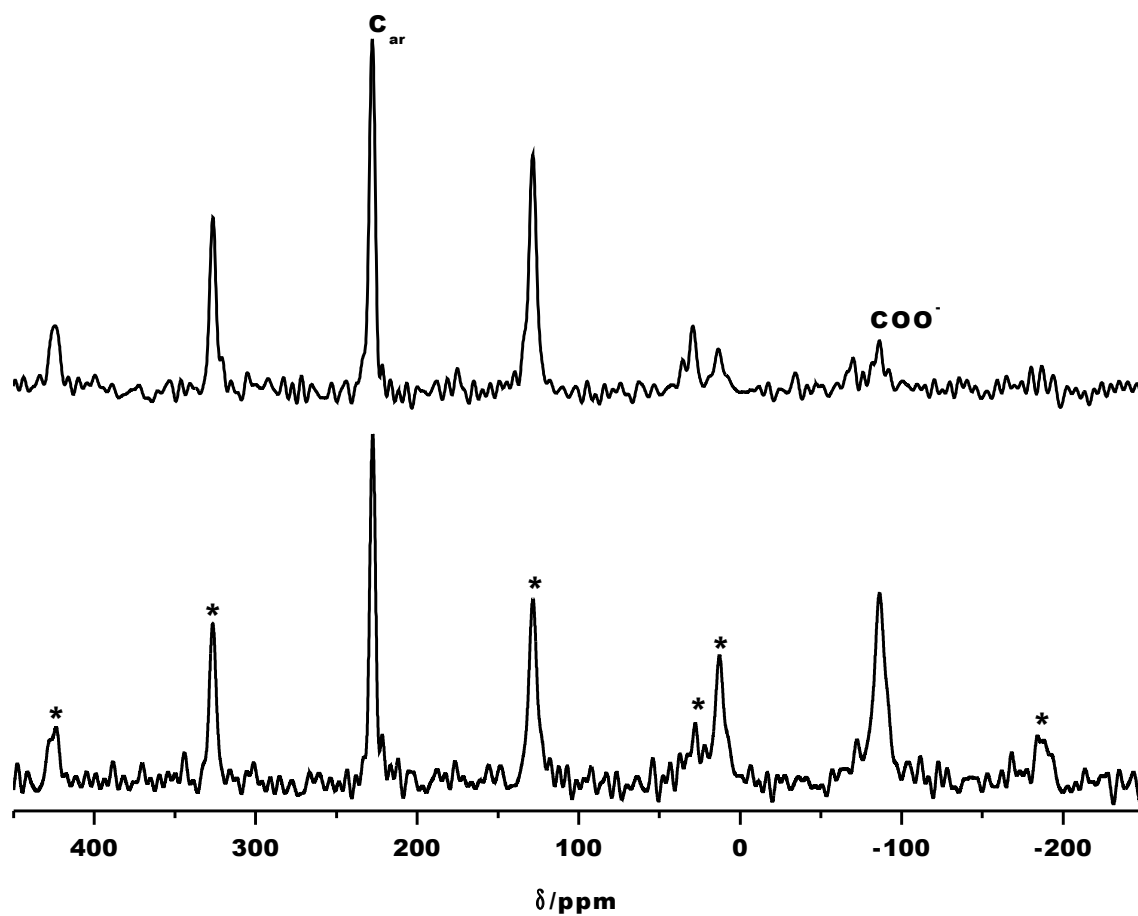
## Supporting Information



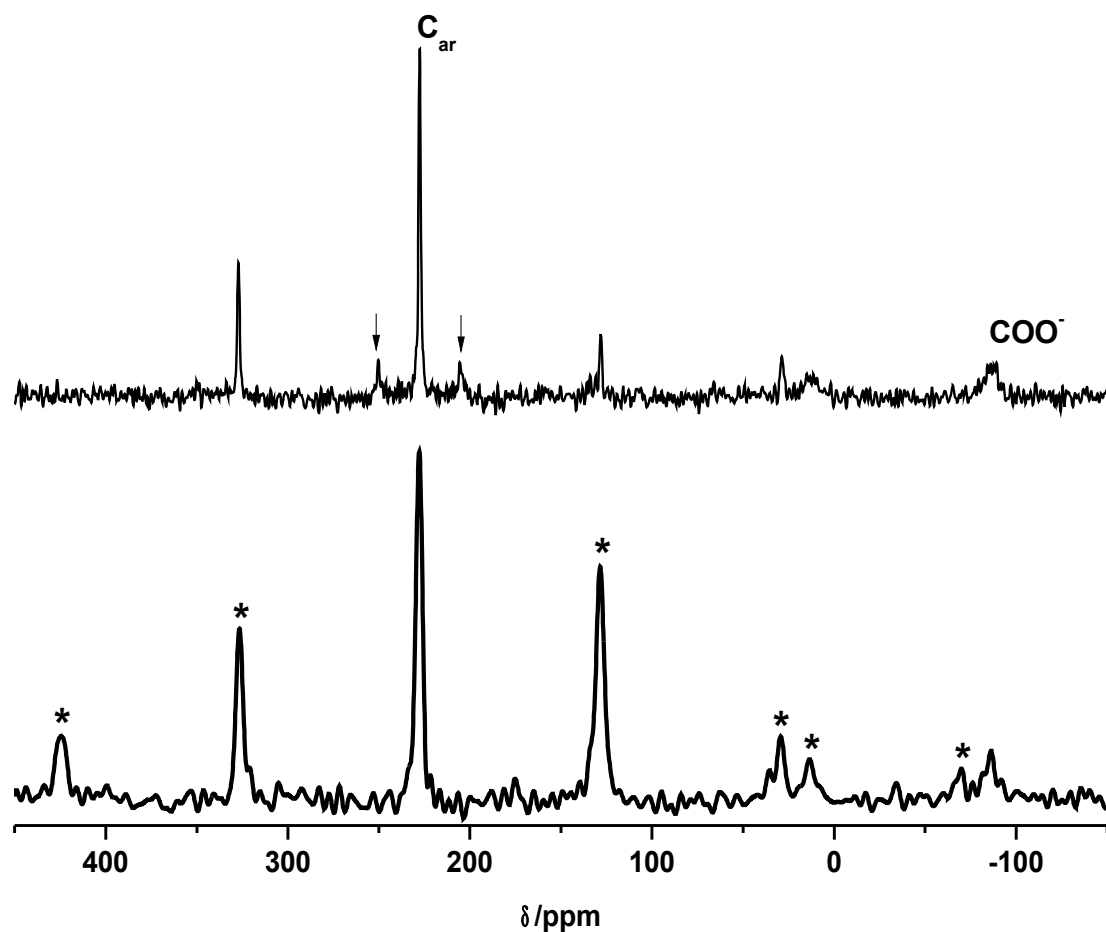
**Figure S-1:** XRD powder pattern of  $\text{Cu}_3(\text{BTC})_2$  (top), and calculated pattern from single crystal X-ray data of  $\text{Cu}_3(\text{BTC})_2$  (bottom) at 293 K.



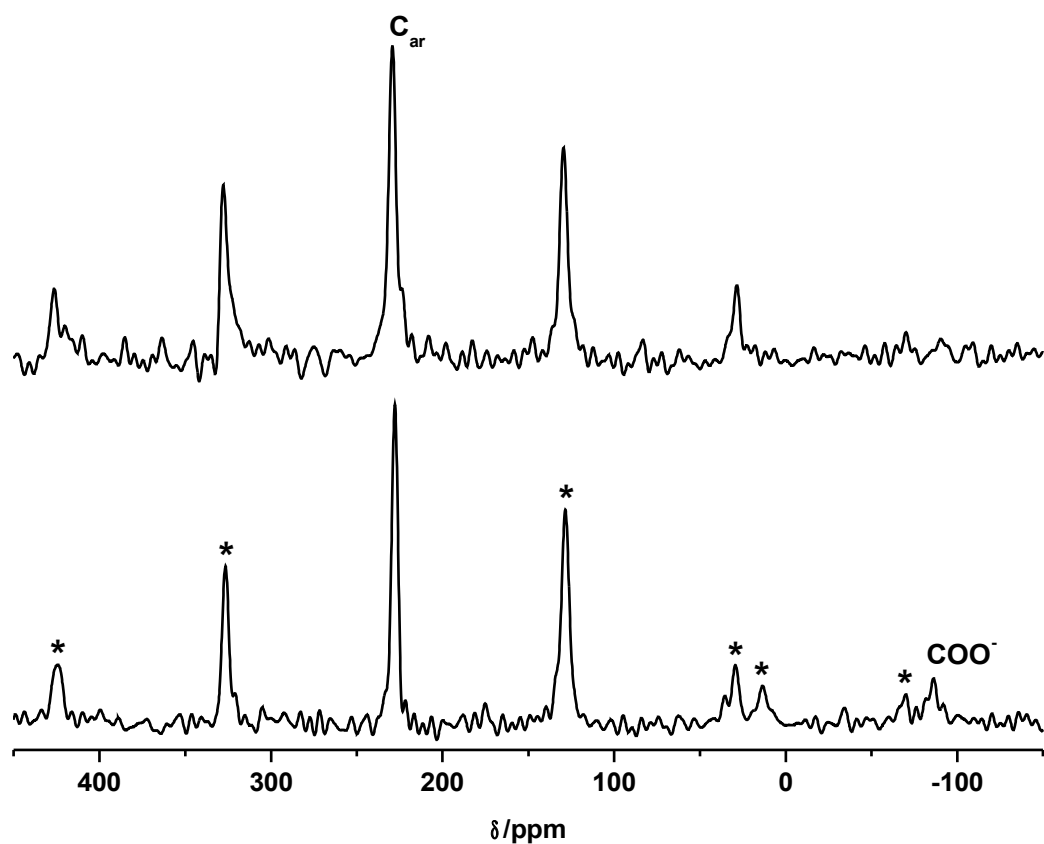
**Figure S-2:**  $^{13}\text{C}$  DPMAS spectra of dehydrated  $\text{Cu}_3(\text{BTC})_2$  with (top) and without (bottom) high-power proton decoupling. Additional signals indicated by arrows are only visible with  $^1\text{H}$  decoupling. Asterisks (shown only in the lower spectrum) denote spinning sidebands.



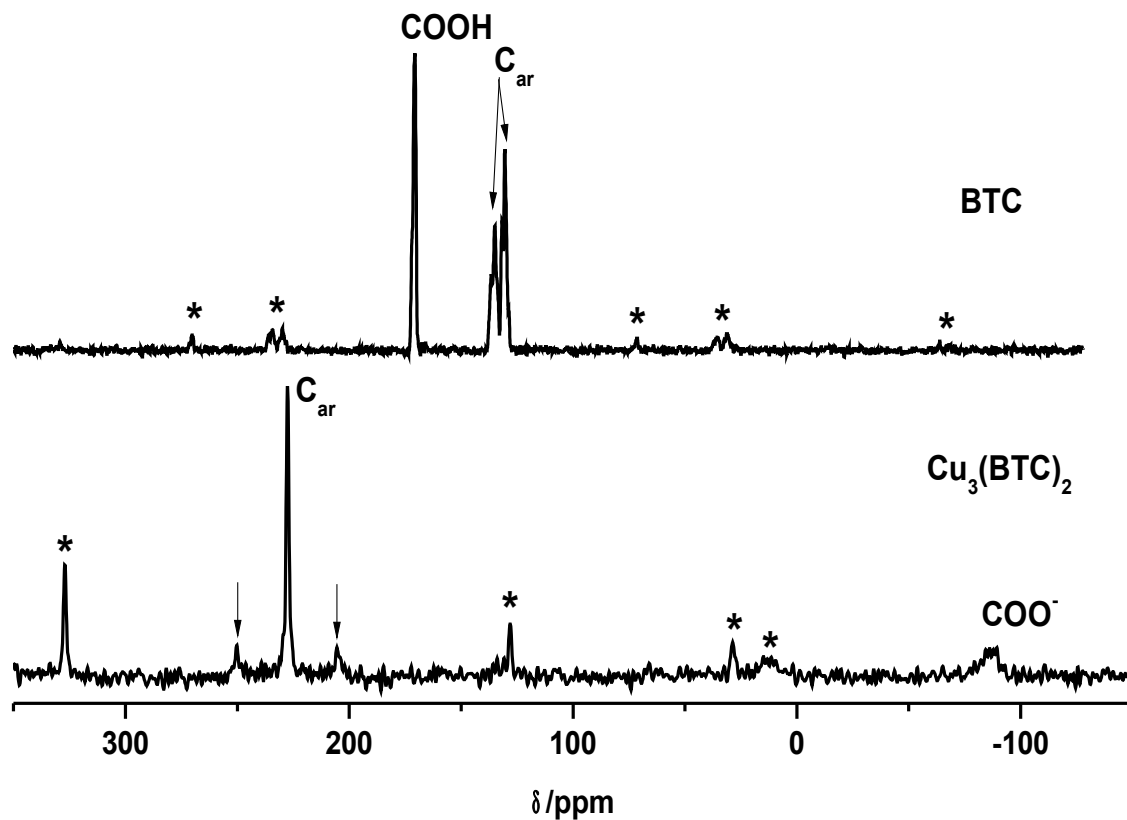
**Figure S-3:**  $^{13}\text{C}$  CPMAS (top) and DPMAS (bottom) spectra of dehydrated  $\text{Cu}_3(\text{BTC})_2$  without high-power proton decoupling. Asterisks (shown only in the lower spectrum) denote spinning sidebands.



**Figure S-4:**  $^{13}\text{C}$  CPMAS spectra of dehydrated  $\text{Cu}_3(\text{BTC})_2$  with (top) and without (bottom) high-power proton decoupling. Additional signals indicated by arrows are only visible with  $^1\text{H}$  decoupling. Asterisks (shown only in the lower spectrum) denote spinning sidebands.



**Figure S-5:**  $^{13}\text{C}$  CPMAS spectra of dehydrated  $\text{Cu}_3(\text{BTC})_2$  with a short contact time of  $50\ \mu\text{s}$  (top) and a long contact time of  $500\ \mu\text{s}$  (bottom). Asterisks (shown only in the lower spectrum) denote spinning sidebands.



**Figure S-6:**  $^{13}\text{C}$  CPMAS spectra of  $\text{Cu}_3(\text{BTC})_2$  (bottom) and the ligand benzenetricarboxylic acid (top) at 10 kHz rotation frequency with high power proton decoupling. Additional signals indicated by arrows are only visible with  $^1\text{H}$  decoupling. Asterisks denote spinning sidebands.