



**S3 Fig. Effect of the derivatization reaction time on observed  $^{12}\text{C}:^{13}\text{C}$  ratio.** (A) Derivatization was carried out on six different  $^{12}\text{C}/^{13}\text{C}$  SCFA standard mixed solutions with a  $^{12}\text{C}:^{13}\text{C}$  concentration ratio of 0.5, 0.8, 1, 1.25, 2 and 5. Aliquots were sampled over time. Dashed line represents a slope = 1. Error bars represent standard deviation, averaged data for the three SCFAs. (B) Regression line equations and  $r^2$  corresponding to plots in (A). (C) MS Signal intensity is plotted over time for the 3 standard isotopologue pairs present in an equimolar ratio in the sample; expressed in arbitrary units (a.u.).