

Standards	Regression line equation	r²
¹² C-Ac	y = 4.24E+06x + 2.29E+06	0.998
¹³ C-Ac	y = 4.38E+06x + 3.04E+06	0.995
¹² C-Prop	y = 7.12E+06x + 5.71E+06	0.997
¹³ C-Prop	y = 8.16E+06x + 6.33E+06	0.996
¹² C-But	y = 1.01E+07x + 8.23E+06	0.998
¹³ C-But	y = 1.06E+07x + 1.04E+07	0.997

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Analyte concentration (µM)	0.02	0.04	0.08	0.16	0.31
Acetate	1.6	3.3	4.0	6.4	10.2
Propionate	2.3	3.6	4.3	10.4	21.9
Butyrate	2.6	3.4	7.0	10.3	14.8

S1 Fig. Calibration curves, LLOD and LLOQ determination for derivatized SCFA standards. (A) Calibration curves for the 3 isotopologue pairs of SCFA standards generated from the derivatization of an equimolar standard solution. Concentrations ranged from 20 nM to 100 μ M. Dotted lines represent ¹³C-standards. Error bars indicate standard deviation, (n =3 technical replicates). MS signal intensities are plotted in arbitrary units (a. u.). (B) Regression line equations and r^2 corresponding to plots in (A). (C) Signal-to-noise ratio (S/N) from chromatographic peaks for 3 analytes (acetate, propionate and butyrate derivatized with aniline) at five different concentrations (0.02, 0.04, 0.08, 0.16 and 0.31 µM, in H₂O/ACN (50:50, v/v)). Bold figures indicate the signal-to noise ratios used for the determination of the LLOD and LLOQ for the analytical method (≥ 3 and ≥ 10 , respectively).

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