Table showing 2019 Instrumental parameters used for MRM analysis. The ion selected for quantification is listed first for each compound. DP: de-clustering potential; EP: entrance potential; CE: collision energy; CXP: collision cell exit potential.

Compound	Instrument setup	Polarity	Q1	Q3	DP	EP	CE	CXP
Organic_A	2	-	145	101	-30	-6	-15	-9
				83			-17	-10
Organic_B	1	+	114	79	81	8	23	5
				69			25	8
				96			21	8
Organic_C	2	+	350	198	51	10	21	18
				322			13	29
				153			18	20
Organic_D	1	-	205	131	-45	-11	-13	-10
				205			-5	-10
*d*4-cholic acid (Internal standard)	1, 2, 3	-	411	411	-210	-7	-24	-10
				347			-49	-15

Table showing 2020 & 2021 Instrumental parameters used for MRM analysis. The ion selected for quantification is listed first for each compound. DP: de-clustering potential; EP: entrance potential; CE: collision energy; CXP: collision cell exit potential.

Compound	Polarity	Parent ion	DP	EP	Daughter ion	CE	CXP
Organic_A	**neg**	**145.05**	**-30**	**-6**	**101**	**-15**	**-9**
					83	**-17**	**-10**
Organic_B	**pos**	**114.0919**	**81**	**8**	**79**	**23**	**5**
					69	**25**	**8**
					96	**21**	**8**
Organic_C	**pos**	**349.93**	**51**	**10**	**198**	**21**	**18**
					153	**18**	**20**
Organic_D	**neg**	**205.1228**	**-45**	**-11**	**161**	**-13**	**-10**
					205	**-5**	**-10**
d4-Cholic acid	**neg**	**411.3048**	**-210**	**-7**	**411**	**-24**	**-10**
					347	**-49**	**-15**

LC-MS instrumental setups:

2019:

Setup 1

A binary gradient with mobile phase A of 5 mM NH_4OAc and with mobile phase B of ACN and 5 mM NH_4OAc was used. The flow rate was set to 0.2 mL/min. The gradient started with 50% B for 2 min and increased to 99% of phase B during 2-2.5 min. The gradient was then kept at 99% B for 3.5 min, followed by equilibrium at 50% B for 5 min.

Setup 2 & 3

A binary gradient with mobile phase A of 5 mM NH_4OAc and with mobile phase B of ACN and 5 mM NH_4OAc was used. The flow rate was set to 0.3 mL/min. The gradient started with 1% B for 2 min and increased to 95% of phase B during 2-6 min. The gradient was then kept at 95% B for 7 min, followed by equilibrium at 1% B for 7 min.

2020 & 2021:

Setup 4

A binary gradient with mobile phase A of 5 mM NH_4OAc and with mobile phase B of ACN and 5 mM NH_4OAc was used. The flow rate was set to 0.3 mL/min. The gradient started with 30% B for 2 min and increased to 95% of phase B during 2-6 min. The gradient was then kept at 95% B for 7 min, followed by equilibrium at 30% B for 7 min.

Table showing The electrospray ionization (ESI) parameters for the three instrument setups.

Setups	Curtain gas	Needle current/ ion spray voltage	Source temperature	GS1	GS2
1st	35	5.5 kV (+) / -4.5 kV (-)	550 °C	50	60
2nd	35	5.5 kV (+) / -4.5 kV (-)	500 °C	60	65
3rd	35	5.5mA (+) /-4.5 mA (-)	500 °C	60	65