

## Supporting Information

### Rapid chemical analysis of bear bile: a 5-minute separation and quantitation of bile acids using UHPLC/qTOF-MS

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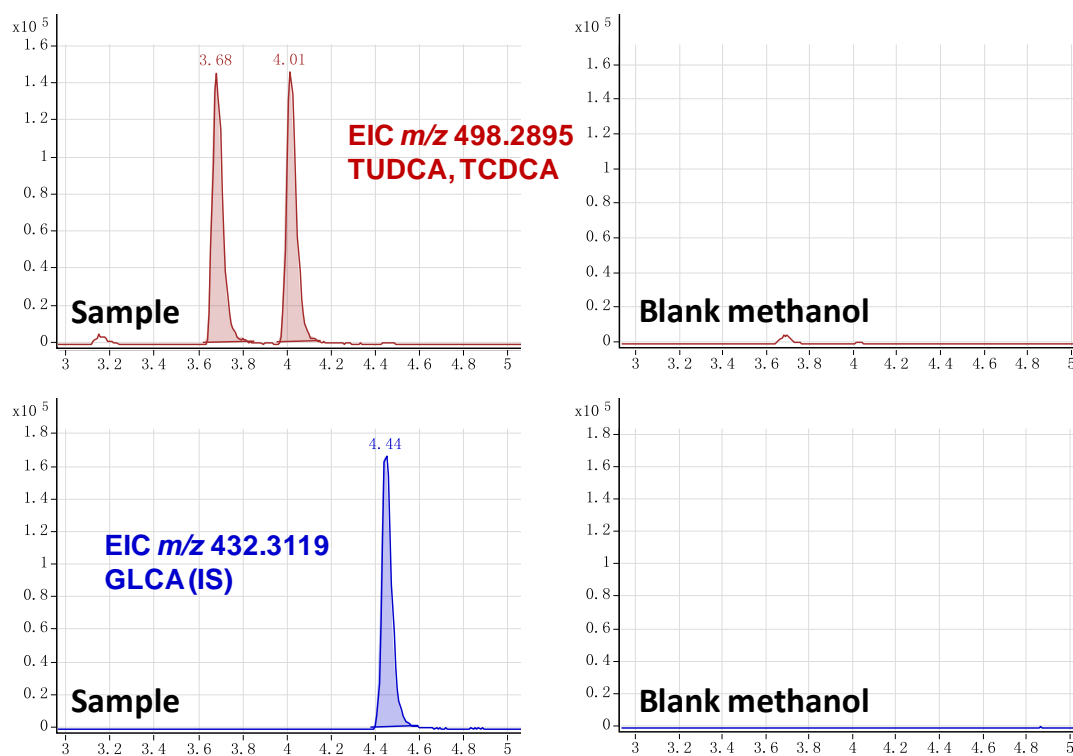
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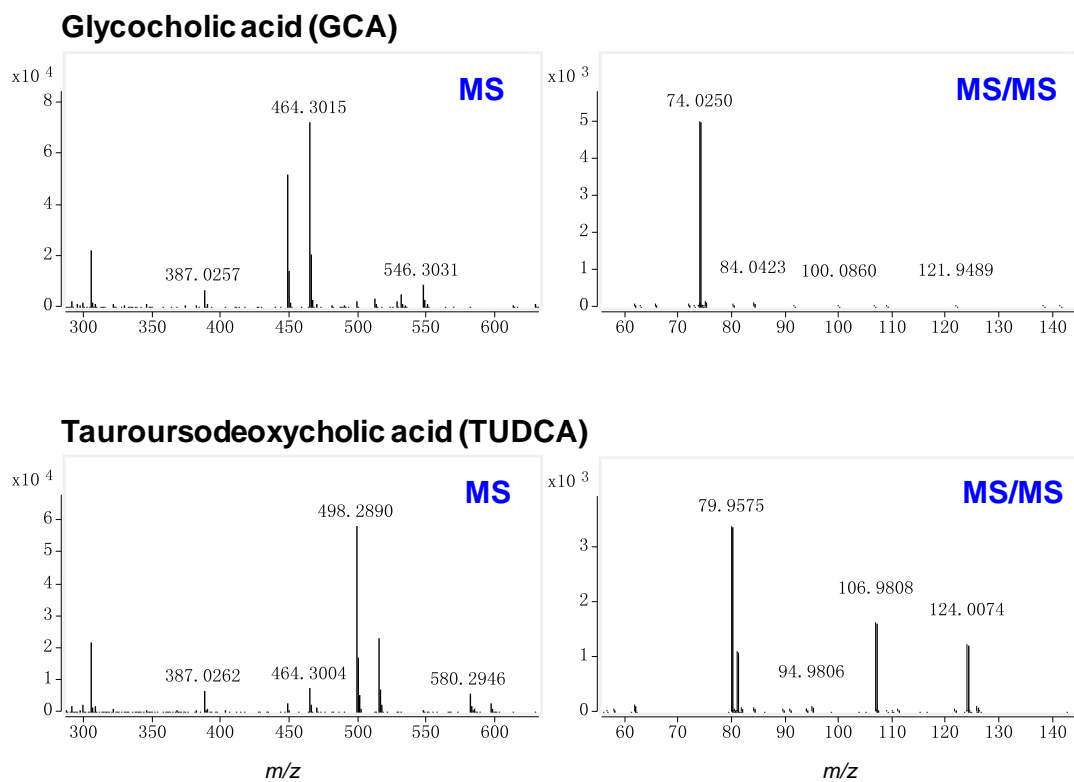
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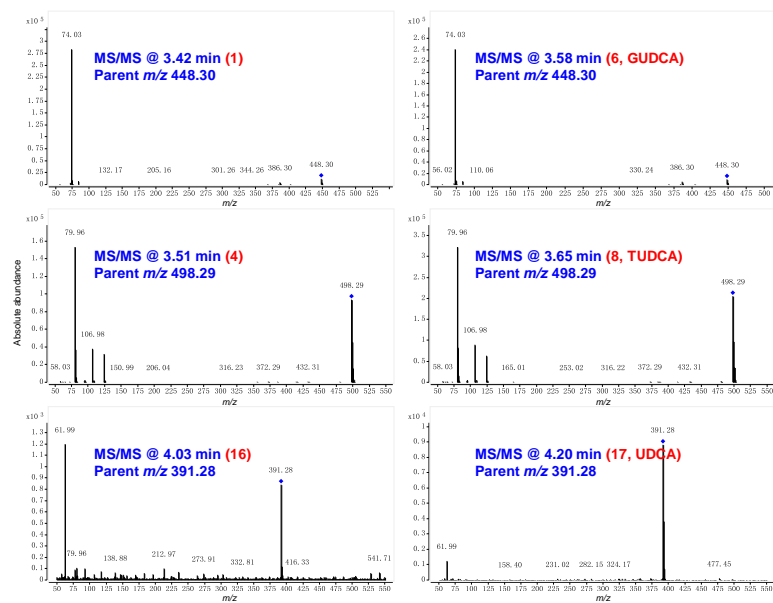
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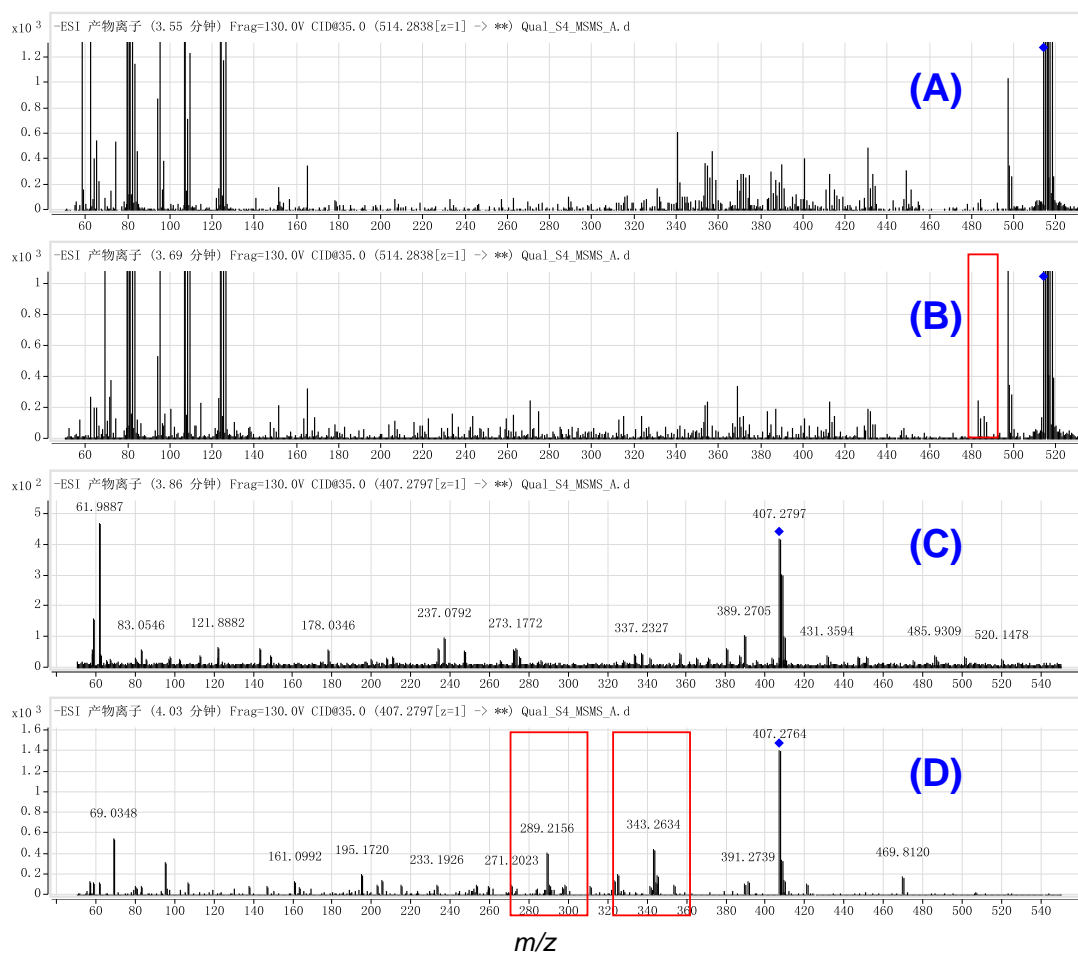
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**Figure 4S.** Minor MS/MS product ions of compounds **5** (A), **9\*** (B), **11** (C) and **13\*** (D).

**Table 1S.** Intraday and interday variation of the analytical method.

Analyte	Concentration ( $\mu\text{g/mL}$ )	Intraday Precision		RE	Interday Precision		RE
		RSD%	( $n=6$ )	%	RSD%	( $n=3$ )	%
TUDCA	LLOQ (0.08)	1.45		-3.53	1.87		-2.58
	MQC (0.25)	2.02		6.71	1.23		4.21
	HQC (0.75)	1.59		1.08	1.95		2.51
TCDCA	LLOQ (0.08)	5.35		4.64	2.32		0.54
	MQC (0.25)	1.66		-2.98	1.53		-5.60
	HQC (0.75)	0.74		-7.39	1.12		-7.73

Note: RSD, relative standard deviation; RE, Relative error (calculated by (measured concentration / nominal concentration - 1)  $\times$  100%).

**Table 2S.** Recovery of bile acid analytes in quantitative analysis.

Analyte		Spiked ( $\mu\text{g/mL}$ )	Found ( $\mu\text{g/mL}$ )	Recovery (%)
TUDCA	LLOQ	0.0400	0.0423	105.75
	MQC	0.3750	0.3542	94.45
	HQC	3.7500	3.7756	100.68
TCDCA	LLOQ	0.0400	0.0381	95.25
	MQC	0.3750	0.3943	105.15
	HQC	3.7500	4.0045	106.79

Note: Recovery (%) = concentration found / concentration spiked  $\times$  100%.



**Table 3S.** Stability of the analytes after ambient and refrigerated storage.

Analyte	Nominated conc. ( $\mu\text{g/mL}$ )	RE% (Ambient 24 h)	RE% (Ambient 36 h)	RE% (-20 °C 48 h)
TUDCA	LLOQ (0.08)	10.05	3.42	12.40
	MQC (0.25)	-2.25	-3.31	7.59
	HQC (0.75)	1.20	1.34	3.59
TCDCA	LLOQ (0.08)	-3.34	-4.60	2.76
	MQC (0.25)	-3.07	-1.96	-13.30
	HQC (0.75)	-2.23	-0.77	1.39

Note: RE, Relative error (calculated by (measured concentration / nominal concentration - 1)  $\times$  100%).

**Table 4S.** Occurrence of major and minor bile acids in 11 commercial bear bile samples.

Sample	Compound No.																				
	1	2	3	4	5	6*	7	8*	9*	10	11	12*	13*	14*	15	16	17*	18	19*	20	21*
1	-	-	+	-	-	-	+	+	+	+	-	-	-	+	+	-	-	+	+	-	-
2	-	-	-	-	-	-	-	+	+	+	+	-	+	+	+	-	+	+	+	+	+
3	-	-	-	-	-	-	-	+	+	+	+	-	+	+	+	-	+	+	+	+	+
4	+	+	-	+	+	+	-	+	+	+	+	+	+	+	+	+	+	+	+	+	+
5	-	-	+	-	-	-	+	+	+	+	-	-	+	+	+	-	-	+	+	+	+
6	-	-	+	-	-	-	+	+	+	+	-	-	-	+	+	-	-	+	+	+	-
7	-	-	-	-	-	-	-	+	+	+	-	-	-	+	+	-	-	+	+	+	-
8	-	-	+	-	-	-	+	+	+	+	-	-	-	+	+	-	-	+	+	+	-
9	-	-	+	-	-	-	-	+	+	+	+	-	+	+	+	+	+	+	+	+	+
10	-	-	+	-	-	-	-	+	+	+	+	-	+	+	+	-	-	+	+	+	+
11	-	-	-	-	-	-	-	+	+	+	-	-	-	+	+	-	-	-	-	+	-

Note: Sample number was in accordance with Table 1; +, detected; -, not detected.