

SUPPORTING INFORMATION

Untargeted Screening EPA/DHA Structured Phospholipids In Krill Oil by Chain Locked Driven Hydrophilic Interaction Liquid Chromatography Tandem Mass Spectrometry

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MATERIALS AND METHODS

Optimization of flow rate. HILIC is a kind of normal phase LC, therefore the phospholipids could be separated class-dependently. To completely separate the target analytes and make the peak sharp and symmetry, the flow rate was optimized from 0.2 to 0.8 mL·min⁻¹. A slower flow rate (0.2 to 0.4 mL·min⁻¹) resulted in broad peak width and extended retention time (the last peak) up to 25 min. A faster flow rate (0.8 mL·min⁻¹) led to a partial overlap of the peaks of PC and PE. Besides, the signals were also influenced because too much solvents were introduced into the MS instrument at the moment. Finally, a flow rate of 0.6 mL·min⁻¹ was selected, under which the phospholipid classes could be well separated on the HILIC column. The phospholipids were ionized with solvents fully evaporated under electrospray ion source.

Table S1. The Optimized DP and CE for PL_{EPA/DHA}.

Phospholipids	DP (eV)	CE (eV)	Phospholipids	DP (eV)	CE (eV)
PC _{EPA}	70	50	PC _{DHA}	100	50
PE _{EPA}	70	40	PE _{DHA}	100	40
PI _{EPA}	70	50	PI _{DHA}	70	50

Table S2. The Validation of PreIS-HILIC-MS/MS Method in Terms of Linearity, Sensitivity, Precision, and Recovery.

Reference materials	Linearity		Sensitivity			Precision		Recovery (% RSD)
	Calibration curve	R ²	Linear range (µg·L ⁻¹)	LOD (µg·L ⁻¹)	LOQ (µg·L ⁻¹)	Intraday precision (RSD%)	Interday precision (RSD%)	
PC 16:0/20:5	Y=1.02e5x-680	0.9981	LOQ - 1000	3.98	13.3	3.22	5.17	90.8%, 2.99
PC 18:0/22:6	Y=1.05e5x-460	0.9990		4.02	13.4	3.68	4.69	87.3%, 3.41
PE 16:0/20:5	Y=3.40e5x-1200	0.9993		2.74	9.13	4.12	5.25	83.5%, 3.52
PE 18:0/22:6	Y=3.04e5x-920	0.9989		2.58	8.60	3.65	4.33	91.4%, 3.66
PI 18:1/20:5	Y=1.43e6x+1820	0.9978		1.03	3.43	4.71	5.98	83.6%, 4.36
PI 16:1/22:6	Y=1.06e6x+980	0.9980		1.12	3.73	4.19	6.24	78.9%, 5.25

Figure S1. The PIS Spectra of (A) PC 18:0/20:5, (B) PE 16:0/20:5, and (C) PI 18:1/20:5.

