Supplementary materials - Carazo et al. 2021, Nutrients

Table S1. Detailed summary of methods for determination of retinoids and carotenoids in human biological materials

Technique	Sensitivity (nmol/L)	Matrix	Analytes	Advantages	Disadvantages	Reference
HPLC-UV/VIS/PDA	[1]5.43 – 49.93	serum	9 carotenoids	* combination with SPE * 9 analytes in 17 min	* usage of toxic solvents (THF)	[1]
	[2]20 – 120	plasma	retinol, carotenoids, 10 analytes	* single LLE * usage only 200 μL of sample	* complicated gradient elution in 32 min	[2]
	[1]0.2 – 2	serum, seminal plasma	retinol, ATRA, carotenoids	* very simple sample preparation * various body fluids * only 250 μL * 15 analytes in 30 min	* complicated gradient elution	[3]
	[2]4.52 – 8.73	plasma	retinol, retinyl esters (palmitate, stearate, oleate, linoleate)	* automated column switching usage	* 5 analytes in 25 min * complicated mobile phase	[4]
	[2]2.29 – 4.19	mouse embryos and kidney, rat plasma	retinol, retinoic acids, retinyl palmitate, 6 analytes	* automated SPE – HPLC	* 6 retinoids in 42 min * complicated multilinear gradient	[5]
	[2]1.75	serum	retinol	* minimized human intervention * fully automated * 35 min per sample	* 2 mL of human serum * triple SPE, double LLE * 5 analytes in 15 min	[6]
	[1]3.49	serum	retinol		* complicated sample preparation using large volumes of solvents	[7]
	[2]87.28	serum	retinol	* 100 μL sample	* 3 analytes in 7 min	[8]
	[1]100	dried whole blood spots	retinol	* 2 analytes in 5 min * DBS – 30 μL of sample	* toxic mobile phase	[9]
	[1]4	breast milk	retinol	* 2 analytes in 1.8 min		[10]

	[1]0.1 × 10^{-3} – 0.25 × 10^{-3}	red blood cells	carotenoids	* same sensitivity with DAD as APCI-MS * both used in this study	* 8 analytes in 30 min * toxic mobile phase * large volumes of toxic solvents in sample preparation procedure	[11]
	[2]4 – 2.8	serum	retinol, carotenoids	* 15 analytes in 13 min by isocratic elution * 200 μL of sample * sensitivity	* large volumes of toxic solvents in sample preparation procedure	[12]
	[2]11.01 – 54.01	serum	carotenoids	* 30 analytes in 45 min	* toxic mobile phase * large volumes of sample and toxic solvents in sample preparation procedure	[13]
	[2]69.82	plasma	retinol	* 100 μL of sample * 4 analytes in 6 min		[14]
	[3]104.73	plasma	retinol	* 3 analytes in 4 min * 200 μL of sample		[15]
	[1]1.5	seminal plasma	retinol	* 200 μL of sample	* 4 analytes in 13 min * large volumes of toxic solvents in sample preparation procedure	[16]
	[2]209.46 [2]33.51 – 37.25	adipose tissue, plasma	retinol carotenoids	* toxic mobile phase, but in comparison with other methods low consumption per analysis * 250 μL of sample		[17]
HPLC-FLD	[1]34.91	plasma	retinol, 2 analytes	* miniaturized SPE in syringe using nanofibers * 300 μL sample	* 2 analytes in 17 min	[18]
	[1]2.3	breast milk, serum	retinol, 2 analytes	* 2 analytes in 1.4 min		[19]
LC-MS	[1]10	urine	retinol, 2 analytes	* 2 analytes in 4 min * simple sample preparation * MRM	* low retinol recovery	[20]
	[1]7.51	plasma	retinol, 3 analytes	* miniaturized SPE, DBS * 100 μL of sample * MRM	* 4 analytes in 10 min	[21]
	[3]261.83	plasma amniotic fluid	retinol	* simple sample preparation	* 5 analytes in 15 min	[22]

	[1]195.49	tears, serum	retinol, 7 analytes	*	tears and blood		[23]
	[3]76.80	serum	retinol, 4 analytes	*	Unisprey as ionisation source less prone to ME - sensitivity without complex sample preparation		[24]
				*	MRM		
	[3]2 – 40 × 10^{-6}	serum	retinoic acids	*	high sensitivity	* 5 analytes in 14.5 min * large volumes of toxic solvents in sample preparation procedure with poor recovery of hydroxylated metabolites of RA	[25]
SFC-MS	[2]14.90 – 70.31	whole blood	15 carotenoids and apocarotenoids	*	15 analytes in 20 min	* 4 carotenoids quantified	[26]
				*	online SFE		
				*	only 10 μL of sample		
	[2]0.36 – 7.03	plasma	carotenoids	*	14 analytes in 10 min		[27]
				*	robotic SLE		
				*	200 μL of sample		
				*	green mobile phase for carotenoids separation		
	[1]0.09 – 0.19 × 10^{-6}	serum	carotenoids and epoxycarotenoids	*	13 analytes in 15 min	* expensive standards of epoxycarotenoids	[28]
				*	100 μL of sample	* toxic solvents used in sample preparation	
						* epoxycarotenoids not quantified	
	[2]27.4 – 54.3	colostrum	7 carotenoids and 25 apocarotenoids	*	32 analytes in 30 min	* apocarotenoids not quantified	[29]
				*	20 μL of sample		
HPLC-ECD	[2]0.67 – 2.81	serum	retinol, ATRA, 13-cis-RA retinyl acetate, retinal	*	6 analytes in 12 min		[30]
				*	sensitivity		
				*	200 μL of sample		
	[1]314.19	rat plasma	retinol	*	7 analytes in 14 min	* double LLE	[31]
	[1]0.4 × 10^{-3}	serum, cervical tissue	carotenoids	*	sensitivity	* 5 analytes in 30 min but corresponds to the date of publication	[32]
				*	20 μL sample	* toxic solvents	

FIA-CL	[1]80.29	serum	retinol	*	suitable only for pharmaceuticals	*	interferences in biomatrix – necessary to use HPLC – impractical * fivefold LLE	[33]
CE-MEKC-UV	[2]2618.26	serum	retinol			*	3 analytes in 20 min * 1 mL of sample * toxic solutions used in sample preparation * 5 min flushing of the capillary between each run	[34]
Affinity CE	[1]0.02	serum albumins solution	retinol, RA for binding study with serum albumins	*	high sensitivity cell usage	*	8 min flushing of the capillary between each run * not real samples used	[35]
Biosensors	[1]15.3	synthetic plasmatic serum	13-cis-RA			*	applied only at artificial serum	[36]
ELISA kits	[3]100	serum, cell lysates, plasma, tissues	retinol	*	serum, cell lysates, plasma, tissues * range 100 – 10 000 nmol/L	*	necessary to predict concentrations and dilute * for research only * cross reactivity with retinol analogues * time and money consuming for small sample series	[37]
	<[1]173.50	serum, plasma, human liquids	retinol	*	serum, plasma, human liquids * range 431 – 34 910 nmol/L	*	for research only * cross reactivity with retinol analogues * time and money consuming for small sample series	[38]
	[1]0.182	serum, cell lysates, plasma, tissues	retinol	*	serum, cell lysates, plasma, tissues	*	for research only * cross reactivity with retinol analogues * time and money consuming for small sample series	[39]
	[1]0.105	serum, plasma, biofluids, cell lysates	retinol	*	serum, plasma, biofluids, cell lysates * sensitivity	*	for research only * cross reactivity with retinol analogue * time and money consuming for small sample series	[40]

	[1]1.63	serum, plasma, biofluids, cell lysates	retinol	*	serum, plasma, biofluids, cell lysates	* for research only * cross reactivity with retinol analogues * time and money consuming for small sample series	[41]
	[1]1.63	serum, plasma	retinol				[42]
	[1]10	serum, plasma, biofluids, cell lysates	β-carotene	*	serum, plasma, biofluids, cell lysates * 40 μL * range 50 – 10 000 nmol/L	* for research only * cross reactivity with carotenoids analogues * time and money consuming for small sample series	[43]
	[3]279.38	colostrum, cattle whole blood and serum	β-carotene	*	portable On-site quantitative testing * range 279 – 27 938 nmol/L	* total carotenoids * only in animal BM * 400 μL BM * for research only	[44]
HPLC-UV kit	[2]69.8	plasma, serum	retinol	*	simple sample preparation	* 3 analytes in 10 min * expensive for small sample series	[45]
	[3]23.62	plasma, serum	carotenoids	*	7 analytes in 10 min * simple sample preparation * 100 μL of sample	* expensive for small sample series	[46]
	[1]174.5	plasma, serum	retinol	*	simple sample preparation * 250 μL of sample	* 3 analytes in 10 min * expensive for small sample series	[47]
HPLC/UHPLC-UV kit	[2]69.8	plasma, serum	retinol	*	possible to combine with 96well plate format * 96 samples in 30 min * 50 μL of sample * available in UHPLC mode: 3 analytes in 3.5 min	* 3 analytes in 9 min in HPLC mode * expensive for small sample series	[48]
UHPLC-UV kit	[2]139.6	plasma, serum	retinol	*	50 μL of sample	* 3 analytes in 1 min	[49]
Raman spectroscopy	not specified	skin, plasma	carotenoids	*	no sample preparation * noninvasive skin measurement * no toxic waste * correlation with levels in blood measured by HPLC	* autofluorescence (sample dependent) * sensitivity not specified	[50-52]

1 LOD Limit of Detection, 2 LOQ Limit of Quantification, 3 LLOQ Lower Limit of Quantification

APCI Atmospheric Pressure Chemical Ionization; ATRA All-trans Retinoic Acid; BM Biological Material; CE Capillary Electrophoresis; DAD Diode Array Detection; DBS Dry Blood Spot; ECD Electrochemical Detection; ELISA Enzyme-Linked ImmunoSorbent Assay; FIA-CL Flow-Injection Analysis with Chemiluminescence detection; FLD Fluorescence Detection; HPLC High Performance Liquid Chromatography; LC-MS Coupling of Liquid Chromatography and Mass Spectrometry; LLE Liquid-Liquid Extraction; ME Matrix Effects; MEKC Micellar Electrokinetic Chromatography; MRM Multiple Reaction Monitoring; MS Mass Spectrometer; PDA PhotoDiode Array Detection; RA Retinoic Acid; SFC-MS Coupling of Supercritical Fluid Chromatography and Mass Spectrometry; SFE Supercritical Fluid Extraction; SLE Solid Supported Liquid-Liquid Extraction; SPE Solid Phase Extraction; THF Tetrahydrofuran; UHPLC Ultra-High Performance Liquid Chromatography; UV/VIS Ultraviolet/Visible Detection;

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