Date: 2025 -02

Supplementary information on EN 18032 (informative)

Foodstuff — Quick Method for the Analysis of Multiple Highly Polar Pesticides and their Metabolites in Foodstuff Involving Extraction with Acidified Methanol and Measurement by LC- or IC-MS/MS (QuPPe-Method) - Supplementary information on the method

Lebensmittel — Schnellmethode zur Bestimmung mehrerer hochpolarer Pestizide und ihrer Metaboliten in Lebensmitteln nach Extraktion mit angesäuertem Methanol und Messung mittels LC- oder IC-MS/MS (QuPPe-Methode) – Ergänzende Informationen zur Methode

Produit alimentaire — Méthode rapide pour l'analyse de plusieurs pesticides hautement polaires et de leurs métabolites dans les aliments impliquant une extraction avec du méthanol acidifié et une mesure par LC- ou IC-MS/MS (QuPPe-Method) – Informations complémentaires sur la méthode

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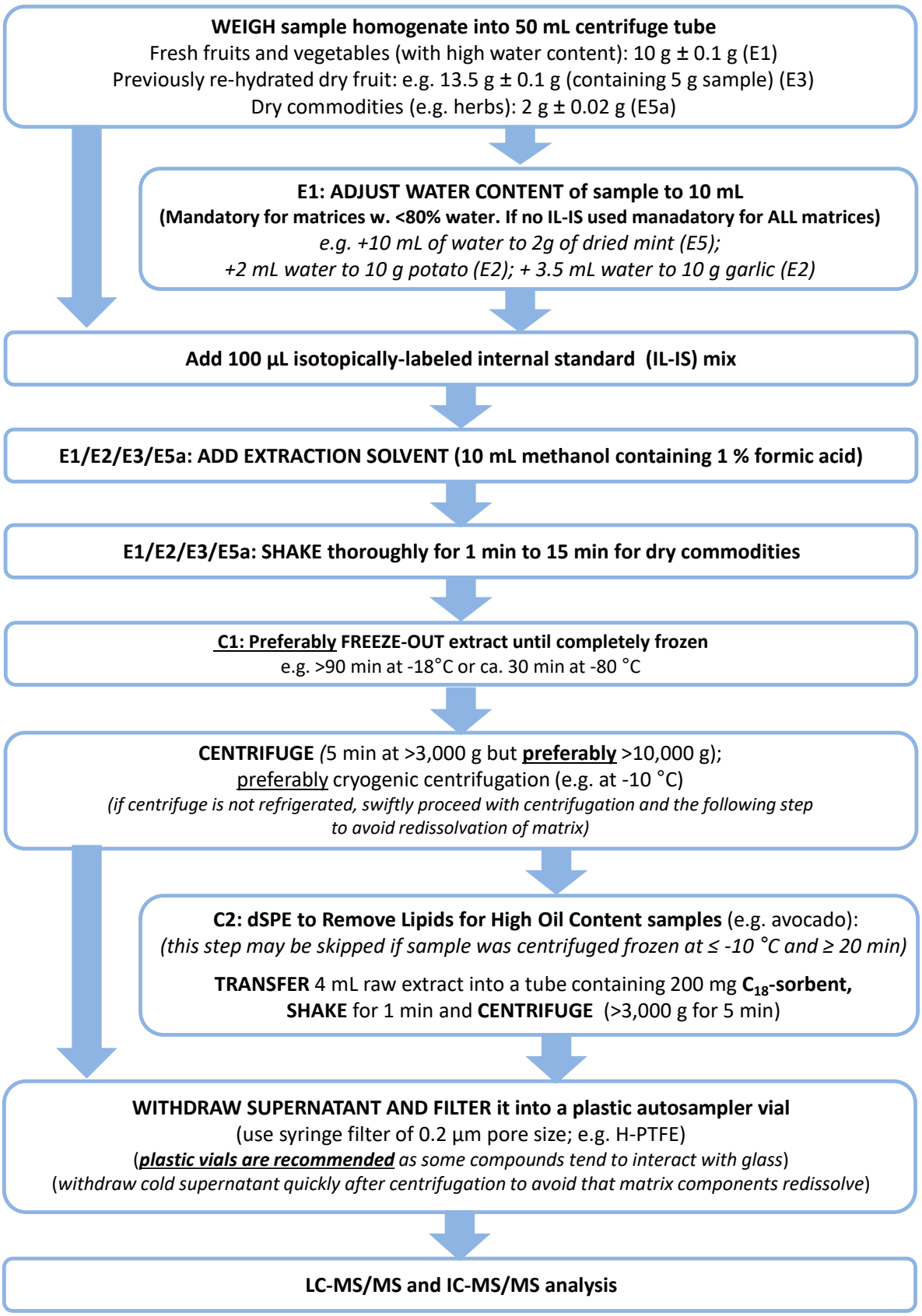
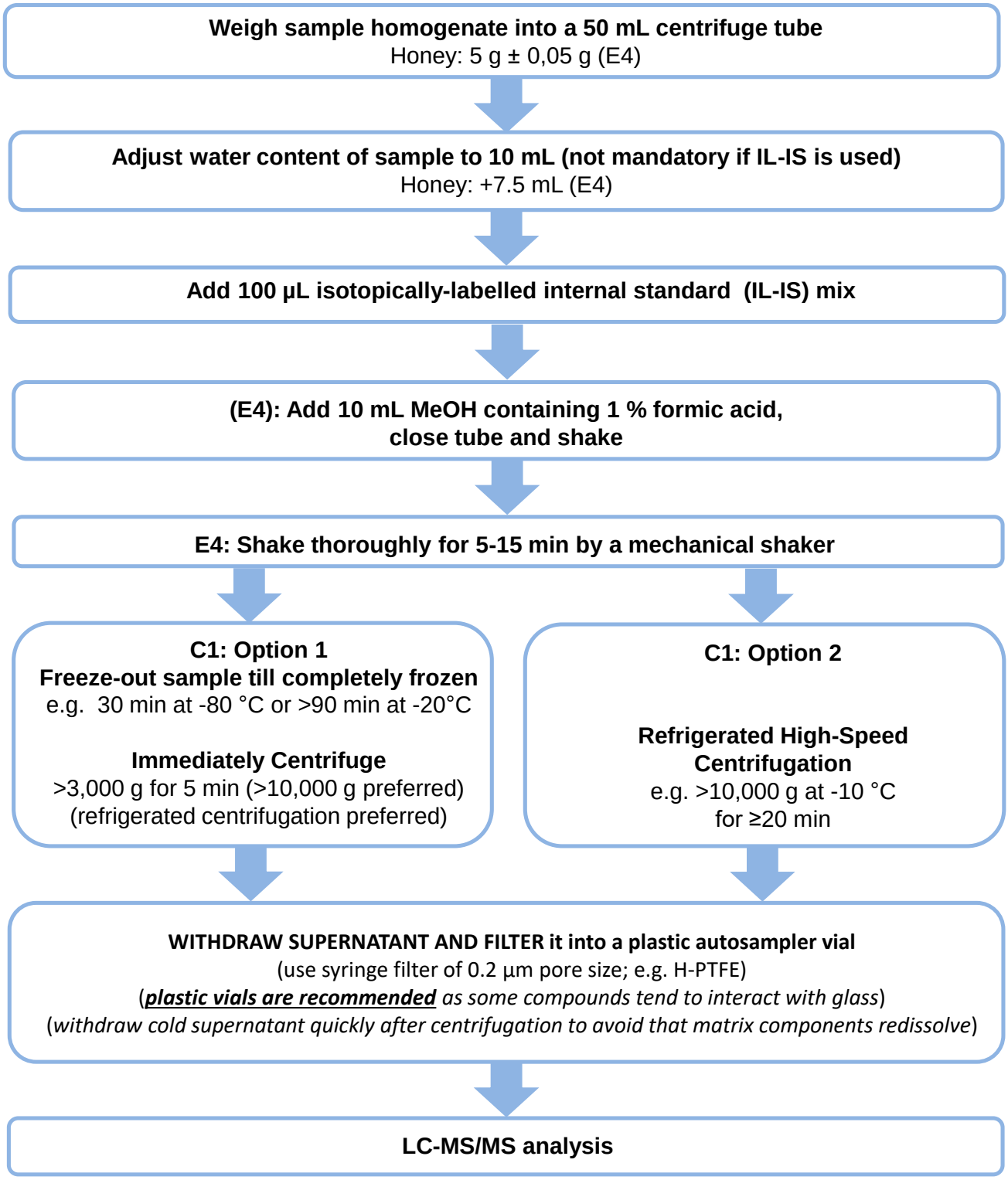
Supplement A (informative):

Schemes showing the method at a glance

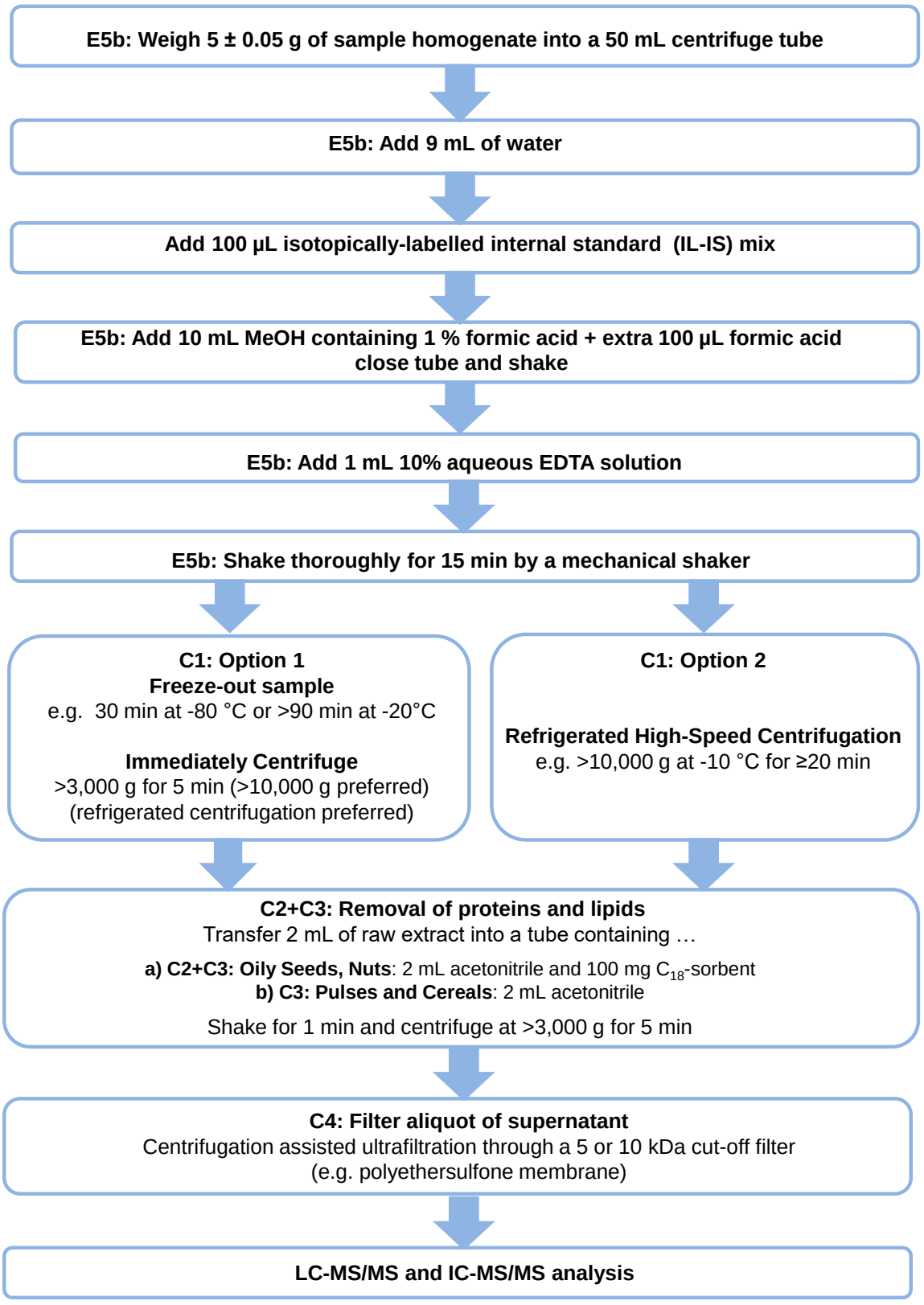
SA.1 QuPPe Method at a glance (for most fruits and vegetables)



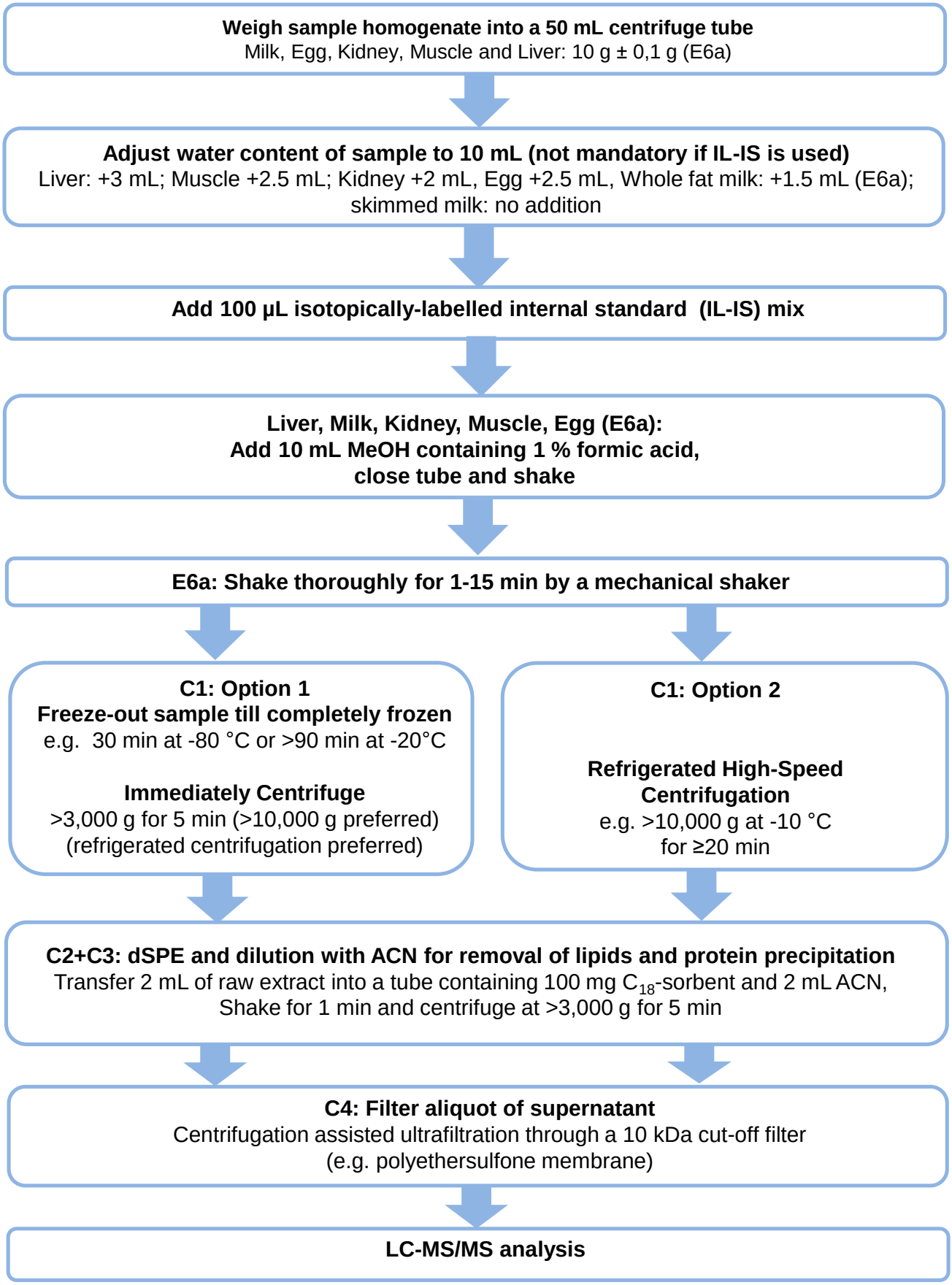
SA.2 QuPPe Method at a glance (for E4 honey)



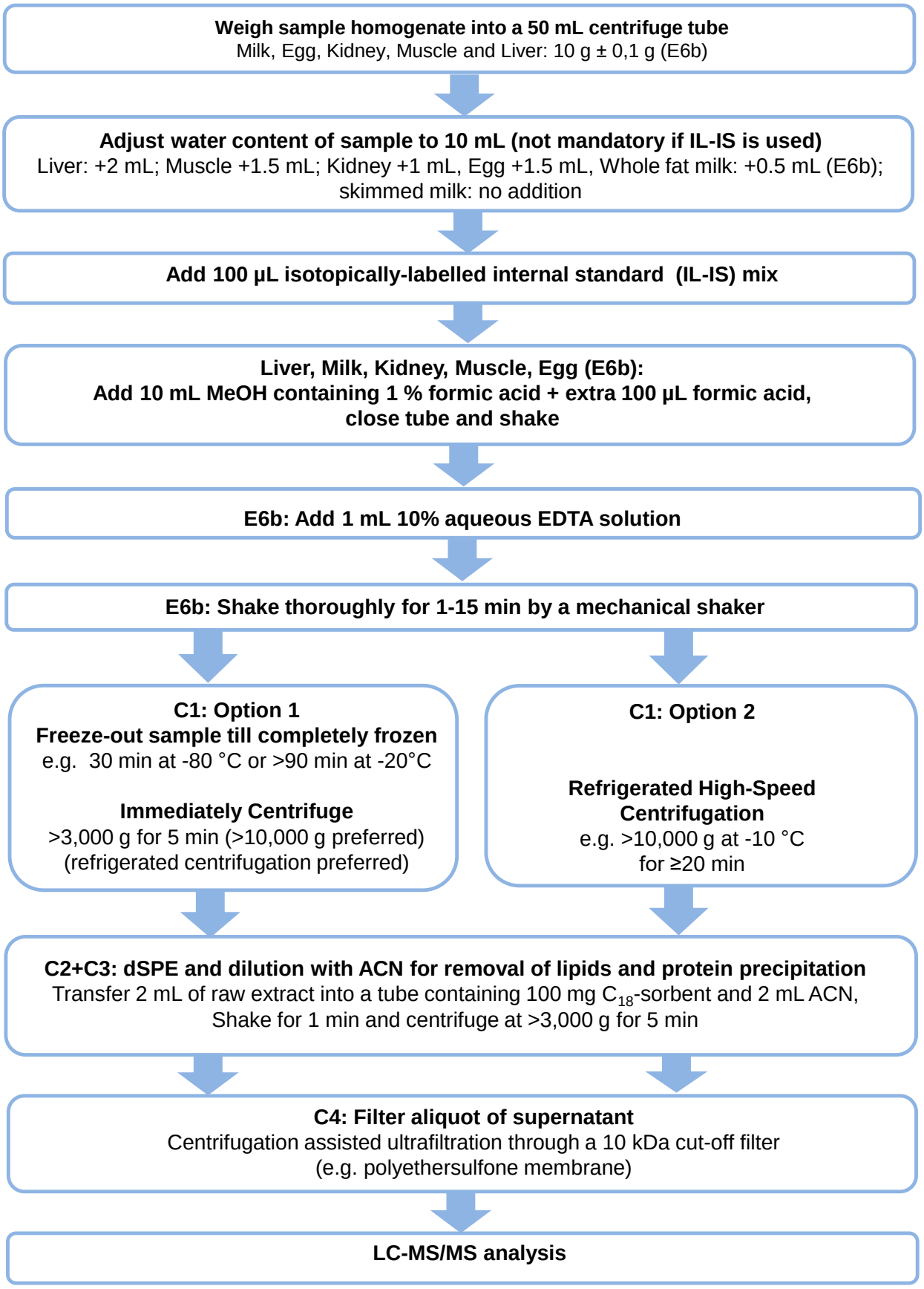
SA.3 QuPPe Method at a glance (for E5b cereals, pulses, nuts and oily seeds)



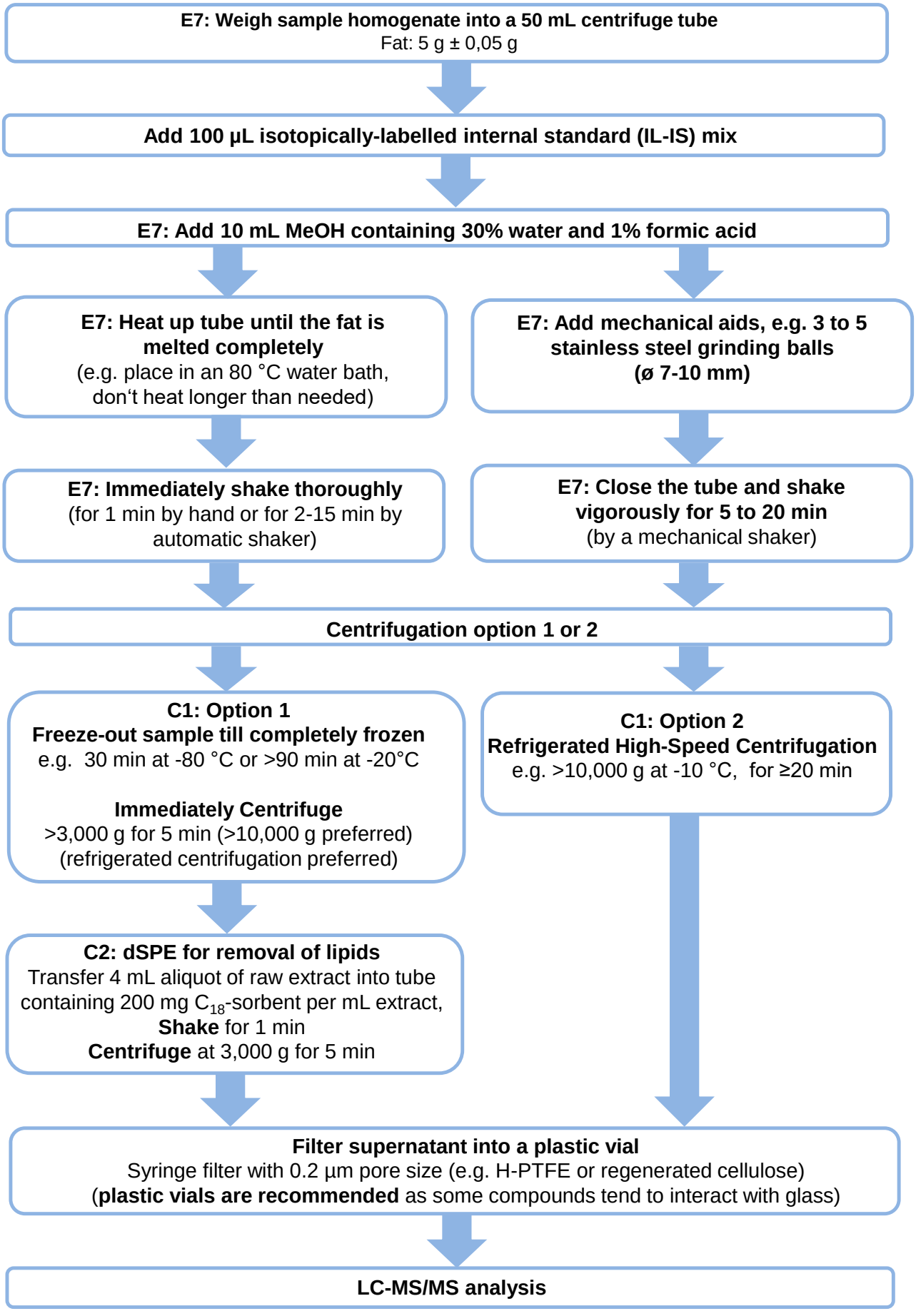
SA.4 QuPPe Method at a glance (for E6a liver, milk, kidney, egg and muscle)



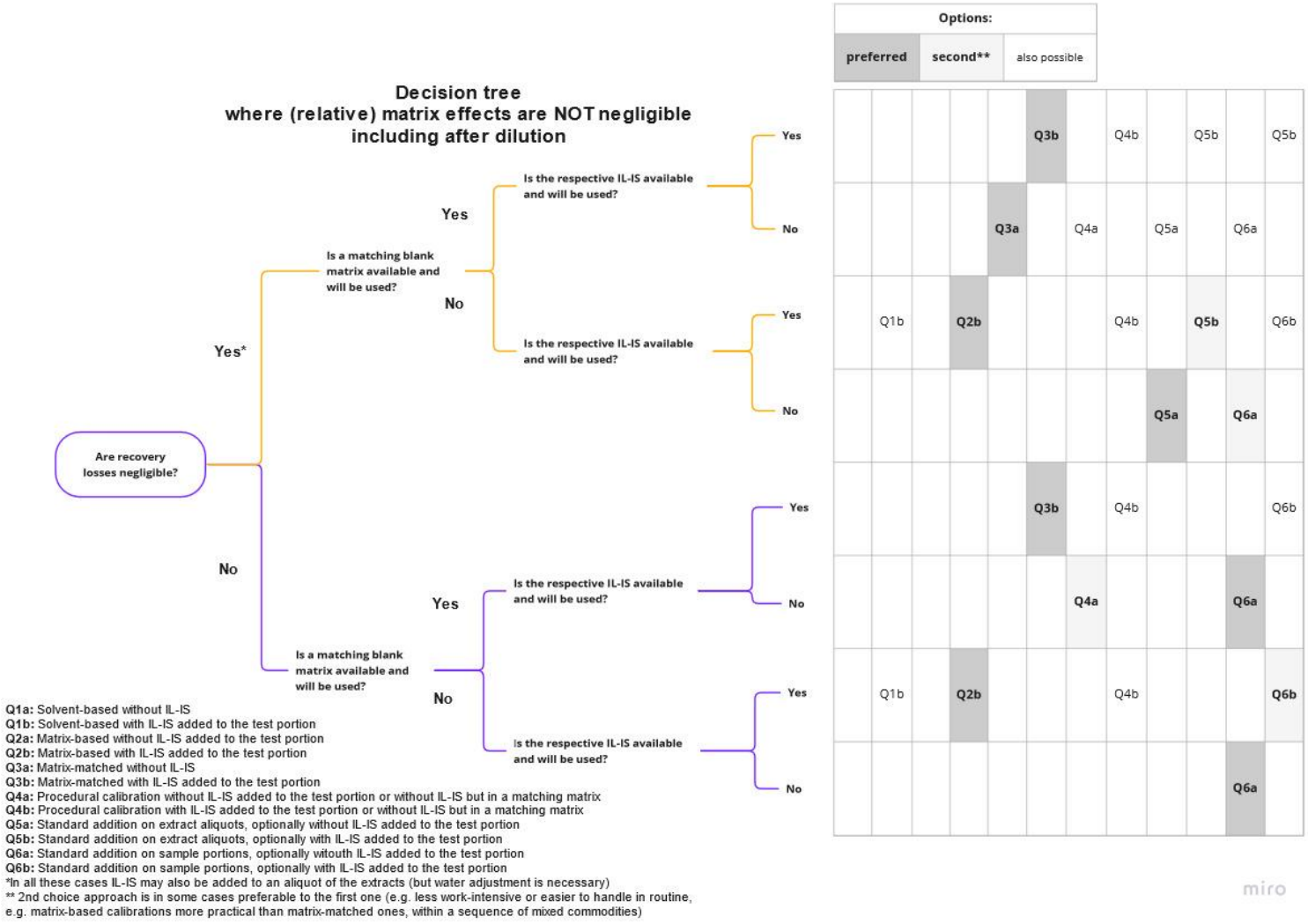
SA.5 QuPPe Method at a glance (for E6b liver, milk, kidney, egg and muscle)

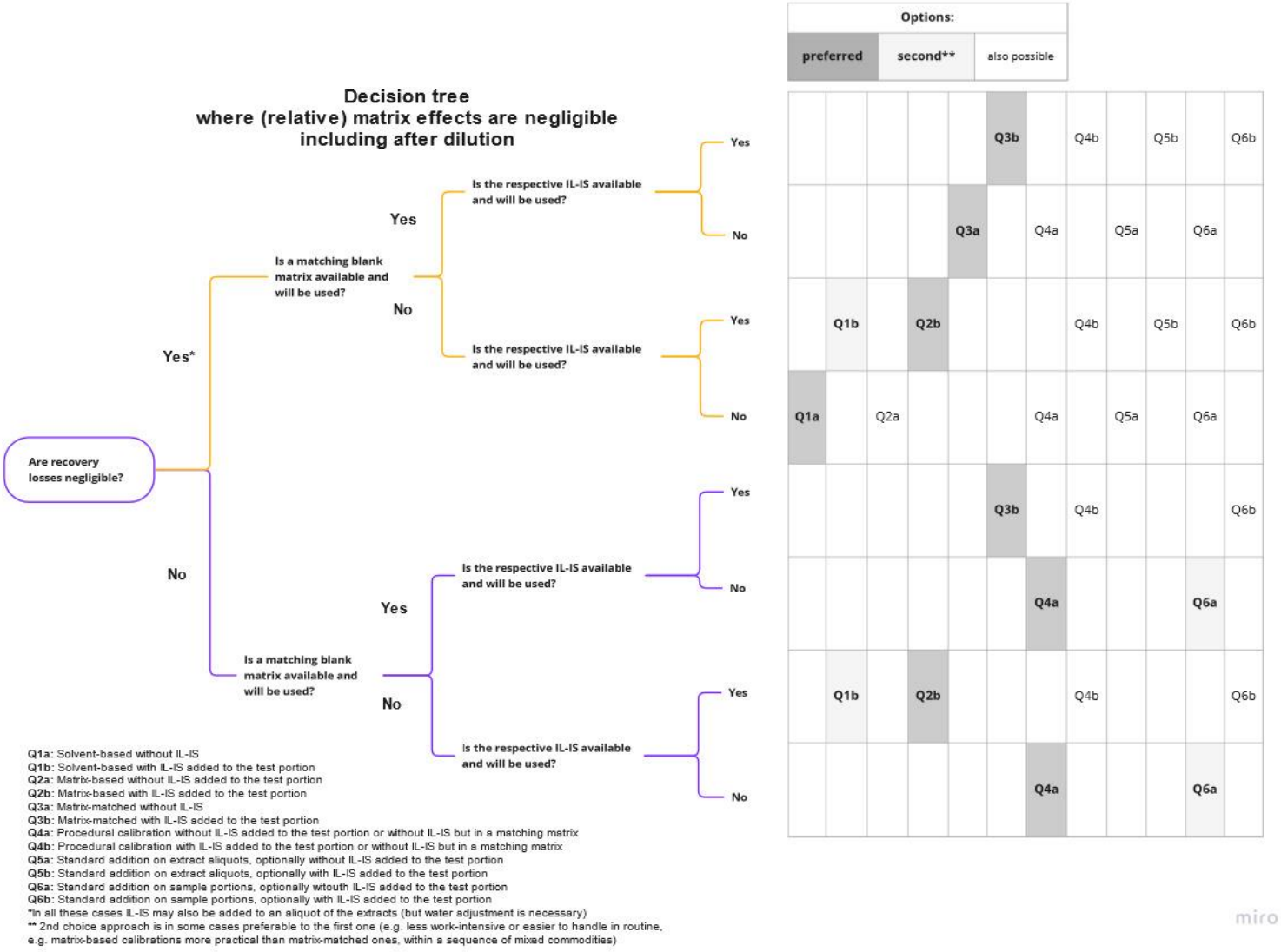


SA.6 QuPPe Method at a glance (for E7 animal fat)



SA.7 Decision trees on the use of quantification modules




Decision tree
where (relative) matrix effects are negligible
including after dilution
Are recovery losses negligible?
Yes*
No
Is a matching blank matrix available and will be used?
Yes
No
Is the respective IL-IS available and will be used?
Yes
No
Is the respective IL-IS available and will be used?
Yes
No
Is a matching blank matrix available and will be used?
Yes
No
Is the respective IL-IS available and will be used?
Yes
No
Is the respective IL-IS available and will be used?
Yes
No
Options:
preferred
second**
also possible
Q3b Q4b Q5b Q6b
Q3a Q4a Q5a Q6a
Q1b Q2b Q4b Q5b Q6b
Q1a Q2a Q4a Q5a Q6a
Q3b Q4b Q6b
Q4a Q6a
Q1b Q2b Q4b Q6b
Q4a Q6a
Q1a: Solvent-based without IL-IS
Q1b: Solvent-based with IL-IS added to the test portion
Q2a: Matrix-based without IL-IS added to the test portion
Q2b: Matrix-based with IL-IS added to the test portion
Q3a: Matrix-matched without IL-IS
Q3b: Matrix-matched with IL-IS added to the test portion
Q4a: Procedural calibration without IL-IS added to the test portion or without IL-IS but in a matching matrix
Q4b: Procedural calibration with IL-IS added to the test portion or without IL-IS but in a matching matrix
Q5a: Standard addition on extract aliquots, optionally without IL-IS added to the test portion
Q5b: Standard addition on extract aliquots, optionally with IL-IS added to the test portion
Q6a: Standard addition on sample portions, optionally witouth IL-IS added to the test portion
Q6b: Standard addition on sample portions, optionally with IL-IS added to the test portion
*In all these cases IL-IS may also be added to an aliquot of the extracts (but water adjustment is necessary)
** 2nd choice approach is in some cases preferable to the first one (e.g. less work-intensive or easier to handle in routine, e.g. matrix-based calibrations more practical than matrix-matched ones, within a sequence of mixed commodities)
miro

Supplement B (informative):

Validation Data

Validation data from interlaboratory validation studies round 1 to 3 is available in tabular form as Excel-Sheet here: QuPPe.eu - EN Supplements

Supplement C (informative):

Additional information

Table SC.1 — Conversion factors between typical purchased standards and target analytes

Compound	MW [g/mol]	Compound as sold	MW [g/mol]	Conversion Factor (CF)	Inverse CF
Bromide (anion)	79,9	Potassium bromide	119,0	0,67	1,49
Chlorate (anion)	83,5	Chlorate-sodium	106,4	0,78	1,27
Chlormequat (cation)[a]	122,6	Chlormequat-chloride[a]	158,1	0,78	1,29
Chlormequat-D_4 (cation)	126,6	Chlormequat-D_4-chloride	162,1	0,78	1,28
Difenzoquat (cation)	249,3	Difenzoquat-methylsulfate	360,4	0,69	1,45
Difluoroacetic acid-$^{13}C_2$	96,0	Sodium difluoroacetate-$^{13}C_2$	120,0	0,80	1,25
Fosetyl	110,0	Fosetyl-Al	118,0[b]	0,93	1,07
Fosetyl-D_5	115,0	Fosetyl-D_5-Al	123,0[b]	0,93	1,07
		Fosetyl-D_5-sodium	137,0	0,84	1,19
Glufosinate	181,1	Glufosinate-ammonium	198,2	0,91	1,09
Glufosinate-D_3	184,1	Glufosinate-D_3-ammonium hydrate	243,2	0,76	1,32
		Glufosinate-D_3-hydrochloride	220,6	0,83	1,20
Mepiquat (cation)[a]	114,2	Mepiquat-chloride[a]	149,7	0,76	1,31
Mepiquat-D_3 (cation)	117,2	Mepiquat-D_3-iodide	244,1	0,48	2,08
Mepiquat-4-hydroxy	130,2	Mepiquat-4-hydroxy-chloride	165,7	0,79	1,27
N-Acetyl-Glufosinate	223,2	N-Acetyl-Glufosinate-disodium	267,2	0,84	1,20
N-Acetyl-Glufosinate-D_3	226,2	N-Acetyl-Glufosinate-D_3-disodium	270,2	0,84	1,19
Nereistoxin	149,3	Nereistoxin-oxalate	239,3	0,62	1,60
Nereistoxin-D_6	155,3	Nereistoxin-D_6-oxalate	245,3	0,63	1,58
Nicotine	162,2	Nicotine hemisulfate	422,5[c]	0,77	1,30
Propamocarb-N-oxide	204,3	Propamocarb-N-oxide hydrochloride	240,7	0,85	1,17
Trifluoroacteic acid	113,0	Sodium trifluoroacetate	136,0	0,83	1,20
Trifluoroacteic acid-$^{13}C_2$	115,0	Sodium trifluoroacetate-$^{13}C_2$	138,0	0,83	1,20
Trimethylsulfonium (cation)	77,2	Trimethylsulfonium-iodide	204,1	0,38	2,64
Trimethylsulfonium-D_9 (cation)	86,2	Trimethylsulfonium-D_9-iodide	213,1	0,40	2,47

[a] Attention: The current EU – MRLs are expressed as the respective chloride salts. Thus, no mathematical conversion of the chloride to the cation is needed.

[b] The MW given here includes only 1/3 of the aluminium mass (one aluminium ion is bound to three fosetyl anions)

c The MW refers to the following formula $(C_{10}H_{14}N_2)_2 \cdot H_2SO_4$ which entails two nicotine molecules.

Table SC.2 — Exemplary concentrations and solvents of stock and working solutions of analytes and their respective IL-ISs

Compound	**Stock Solution (exemplary)**		**Working Solutions including mixtures (exemplary)**	
	Solvent used to prepare	**[mg/mL]**	**Solvent used to prepare**	**[µg/mL]**
Aminocyclopyrachlor	MeOH	1	MeOH	10 / 1 / 0,1
Amitrole	MeOH	1	MeOH	10 / 1 / 0,1
AMPA	10 % ACN in water	1	10 % ACN in water	10 / 1 / 0,1
Bromide	MeOH	1	MeOH	10 / 1 / 0,1 / 0,01
Chlorate	MeOH	1	MeOH	10 / 1 / 0,1 / 0,01
Chloridazon-desphenyl	MeOH	1	MeOH	10 / 1 / 0,1
Chlormequat	MeOH	1	MeOH	10 / 1 / 0,1
Cyanuric acid	MeOH	1	10 % ACN in water	10 / 1 / 0,1
Cyromazine	MeOH	1	MeOH	10 / 1 / 0,1
Daminozide	MeOH	1	MeOH	10 / 1 / 0,1
Difenzoquat	ACN	1	MeOH	10 / 1 / 0,1
Difluoroacetic acid	ACN with 5% water	1	ACN with 5% water	10 / 1/ 0,1
Ethephon	10 % ACN in water + 0,1 % HCl	1	10 % ACN in water + 0,1 % HCl	10 / 1 / 0,1
ETU	MeOH	1	MeOH	10 / 1 / 0,1
Fosetyl	10 % ACN in water	0,1	10 % ACN in water	10 / 1 / 0,1
Glufosinate	10 % ACN in water	1	10 % ACN in water	10 / 1 / 0,1
Glyphosate[b]	10 % ACN in water	1	10 % ACN in water	10 / 1 / 0,1
HEPA	10 % ACN in water	1	10 % ACN in water	10 / 1 / 0,1
Matrine	ACN	1	ACN	10 / 1 / 0,1
Maleic Hydrazide	MeOH	1	10 % ACN in water	10 / 1 / 0,1
Melamine	MeOH:water (90:10)	1	MeOH	10 / 1 / 0,1
Mepiquat	MeOH	1	MeOH	10 / 1 / 0,1
Mepiquat-4-hydroxy	MeOH	1	MeOH	10 / 1 / 0,1
MPPA	10 % ACN in water	1	10 % ACN in water	10 / 1 / 0,1
N-Acetyl-Glufosinate	10 % ACN in water	1	10 % ACN in water	10 / 1 / 0,1
N-Acetyl-Glyphosate	10 % ACN in water	1	10 % ACN in water	10 / 1 / 0,1
Nereistoxin	MeOH / water (3:1)	1	MeOH	10 / 1 / 0,1
Nicotine[b]	ACN	1	ACN	1 / 0,1
Oxymatrine	ACN	1	ACN	10 / 1 / 0,1

Compound	**Stock Solution (exemplary)**		**Working Solutions including mixtures (exemplary)**	
	Solvent used to prepare	**[mg/mL]**	**Solvent used to prepare**	**[µg/mL]**
Perchlorate	MeOH	1	MeOH	10 / 1 / 0,1 / 0,01
Phosphonic acid[b]	Water (^{18}O-H_2O for the ILIS)	1	ACN[c]	10 / 1 / 0,1 / 0,01
Propamocarb	ACN	1	MeOH	10 / 1 / 0,1
Propamocarb-N-desmethyl	ACN:Acetone (1 mL acetone to initially dissolve)	1	MeOH	10 / 1 / 0,1
Propamocarb-N-oxide	MeOH	1	MeOH	10 / 1 / 0,1
PTU	MeOH	1	MeOH	10 / 1 / 0,1
Triazole-lactic acid	MeOH	1	MeOH	10 / 1 / 0,1
Triazole-acetic acid	MeOH	1	MeOH	10 / 1 / 0,1
Triazole-alanine	MeOH / water (1:3)	1	MeOH	10 / 1 / 0,1
Trifluoroacetic acid	ACN with 5% water	1	ACN with 5% water	10 / 1/ 0,1
Trimethylsulfonium	MeOH	1	MeOH	10 / 1 / 0,1

[a] Use plastic vials and protect solutions from light exposure.

[b] Use plastic vessels and stoppers for compounds that tend to interact with glass surfaces.

[c] Pure water (^{18}O-H_2O for the IL-IS) is also suitable for the working solution. 10% ACN will reduce growth of microorganisms.

MeOH: Methanol; ACN: Acetonitrile; FA: Formic acid.