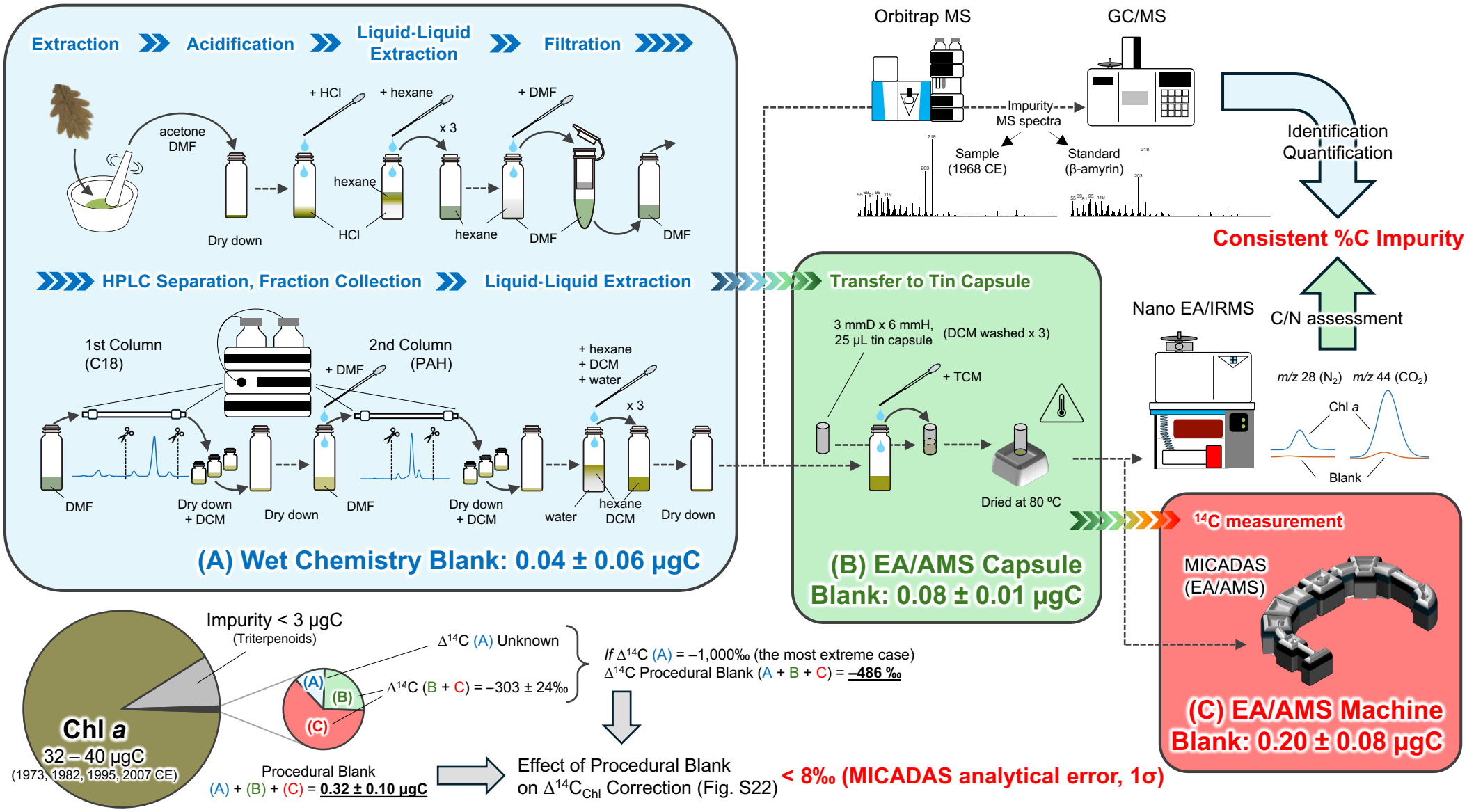
Extraction
Acidification
Liquid-Liquid Extraction
Filtration
acetone
DMF
Dry down
+ HCl
HCl
+ hexane
x 3
hexane
hexane
+ DMF
DMF
DMF
HPLC Separation, Fraction Collection
Liquid-Liquid Extraction
1st Column
(C18)
2nd Column
(PAH)
+ DMF
+ hexane
+ DCM
+ water
x 3
DMF
Dry down
+ DCM
Dry down
DMF
Dry down
+ DCM
water
hexane
DCM
Dry down
(A) Wet Chemistry Blank: 0.04 ± 0.06 µgC
Orbitrap MS
GC/MS
Impurity
MS spectra
Sample
(1968 CE)
Standard
(β-amyrin)
Identification
Quantification
Consistent %C Impurity
Transfer to Tin Capsule
3 mmD x 6 mmH,
25 µL tin capsule
(DCM washed x 3)
+ TCM
Dried at 80 ºC
(B) EA/AMS Capsule
Blank: 0.08 ± 0.01 µgC
Nano EA/IRMS
C/N assessment
m/z 28 (N2)
m/z 44 (CO2)
Chl a
Blank
14C measurement
MICADAS
(EA/AMS)
(C) EA/AMS Machine
Blank: 0.20 ± 0.08 µgC
Impurity < 3 µgC
(Triterpenoids)
Δ14C (A) Unknown
Δ14C (B + C) = −303 ± 24‰
(A)
(B)
(C)
Chl a
32 – 40 µgC
(1973, 1982, 1995, 2007 CE)
If Δ14C (A) = −1,000‰ (the most extreme case)
Δ14C Procedural Blank (A + B + C) = −486 ‰
Procedural Blank
(A) + (B) + (C) = 0.32 ± 0.10 µgC
Effect of Procedural Blank
on Δ14CChl Correction (Fig. S22)
< 8‰ (MICADAS analytical error, 1σ)