Supplementary Information

Cost-effective oxygen flask combustion and electrothermal vaporization capacitively coupled plasma microtorch optical emission spectrometry as green and white method for multielemental determination in food

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This online resource contains the following data:

1. Total carbon, total inorganic carbon and total organic carbon fractions in the solid food samples and absorbing liquid/suspension after oxygen flask combustion
2. Calibration curves obtained by SSETV-μCCP-OES method for different number of integrating pixels of the signal over the spectral line profile
3. Dependence of signal-to-background ratio, relative standard deviation of the background and LODs *versus* the number of integrating pixels of the signal over the spectral line profile
4. Colours of the (OFC)-SSETV-μCCP-OES method in comparison with traditional spectrometric methods

1. **Total carbon, total inorganic carbon and total organic carbon fractions in the solid food samples and absorbing liquid/suspension after oxygen flask combustion**

Table S1. Concentration of total carbon (TC), total inorganic carbon (TIC) and total organic carbon (TOC) in food sample and absorbing liquid/suspension

Sample	Carbon fractions in food sample ± SD[a] (μg mg^{-1})			Carbon fraction in liquid sample/suspension after combustion ± SD[a] (μg mL^{-1})			Combustion efficiency (%)[b]
	TC	TIC	TOC	TC	TIC	TOC	
CRMs							
Tort-3 (Lobster hepatopancreas)	482 ± 15	158 ± 27	324 ± 22	50.2 ± 0.9	19.7 ± 3.0	30.5 ± 3.1	98.1 ± 6.8
CE278k (Mussel tissue)	436 ± 27	66 ± 32	370 ± 17	9.8 ± 0.5	7.3 ± 1.0	2.5 ± 1.1	99.9 ± 4.6
CS-M-3 (Dried mushroom powder)	445 ± 25	19 ± 31	426 ± 19	8.7 ± 0.2	6.2 ± 0.5	2.5 ± 0.5	99.9 ± 4.5
GBW 10011 (Wheat)	395 ± 25	25 ± 34	370 ± 23	10.6 ± 0.9	8.0 ± 0.8	2.6 ± 1.2	99.6 ± 6.2
SRM 3280 (Multivitamin tablets)	129 ± 7	3 ± 9	126 ± 6	15.9 ± 0.9	13.2 ± 0.8	2.7 ± 1.2	99.6 ± 4.8
Food and supplement samples							
Fish tissue 1	537 ± 33	175 ± 39	362 ± 21	20.3 ± 1.3	14.0 ± 1.5	6.3 ± 2.0	99.7 ± 5.8
Fish tissue 2	495 ± 24	177 ± 31	318 ± 19	18.4 ± 1.1	13.1 ± 0.8	5.3 ± 1.4	99.7 ± 6.0
Mushroom 1	409 ± 27	2 ± 37	407 ± 26	8.2 ± 0.9	6.2 ± 0.6	2.0 ± 1.1	99.9 ± 6.4
Mushroom 2	464 ± 30	67 ± 41	397 ± 28	19.3 ± 0.8	16.7 ± 0.9	2.6 ± 1.2	99.9 ± 7.1
Mushroom 3	438 ± 31	34 ± 40	404 ± 25	24.3 ± 1.1	20.2 ± 0.8	4.1 ± 1.4	99.8 ± 6.2
Mushroom 4	451 ± 29	38 ± 38	413 ± 24	15.9 ± 0.8	14.2 ± 0.5	1.7 ± 0.9	99.9 ± 5.8
Supplement 1	693 ± 51	353 ± 53	340 ± 16	9.2 ± 0.8	6.0 ± 0.9	3.2 ± 1.2	99.8 ± 4.7
Supplement 2	636 ± 43	346 ± 46	290 ± 16	8.7 ± 1.1	5.8 ± 0.8	2.9 ± 1.4	99.8 ± 5.5
Supplement 3	164 ± 7	68 ± 8	96 ± 4	8.3 ± 0.6	6.1 ± 0.6	2.2 ± 0.9	99.5 ± 4.2
Pooled combustion efficiency (%)							99.7 ± 5.7

[a]SD is the standard deviation calculated from n = 3 repeated measurements;

[b]Combustion efficiency (%) was calculated as $\frac{m_{TOC\ food\ sample} - m_{TOC\ suspension}}{m_{TOC\ food\ sample}} \times 100$ for 50 mg sample and 500 mL flask combustion.

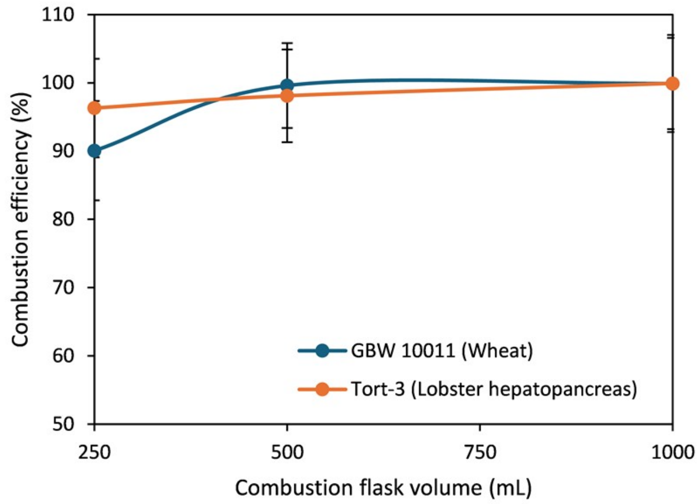
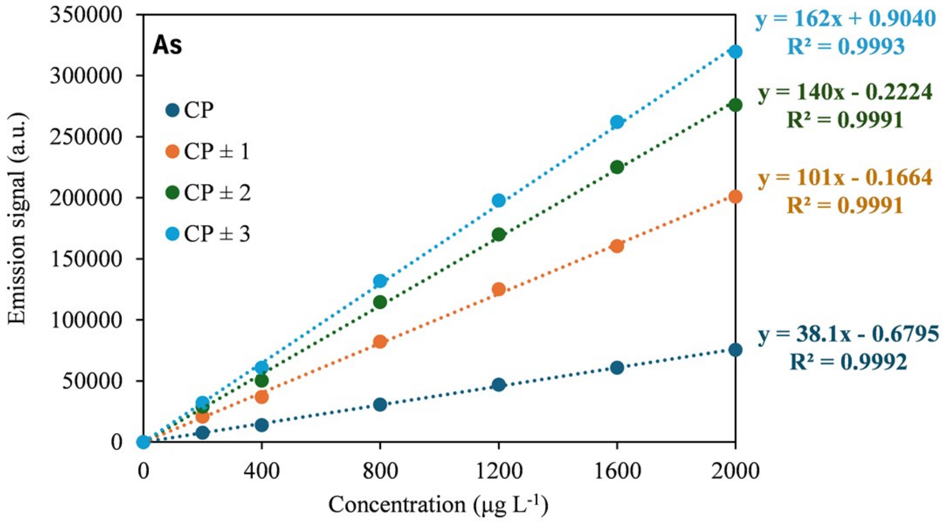
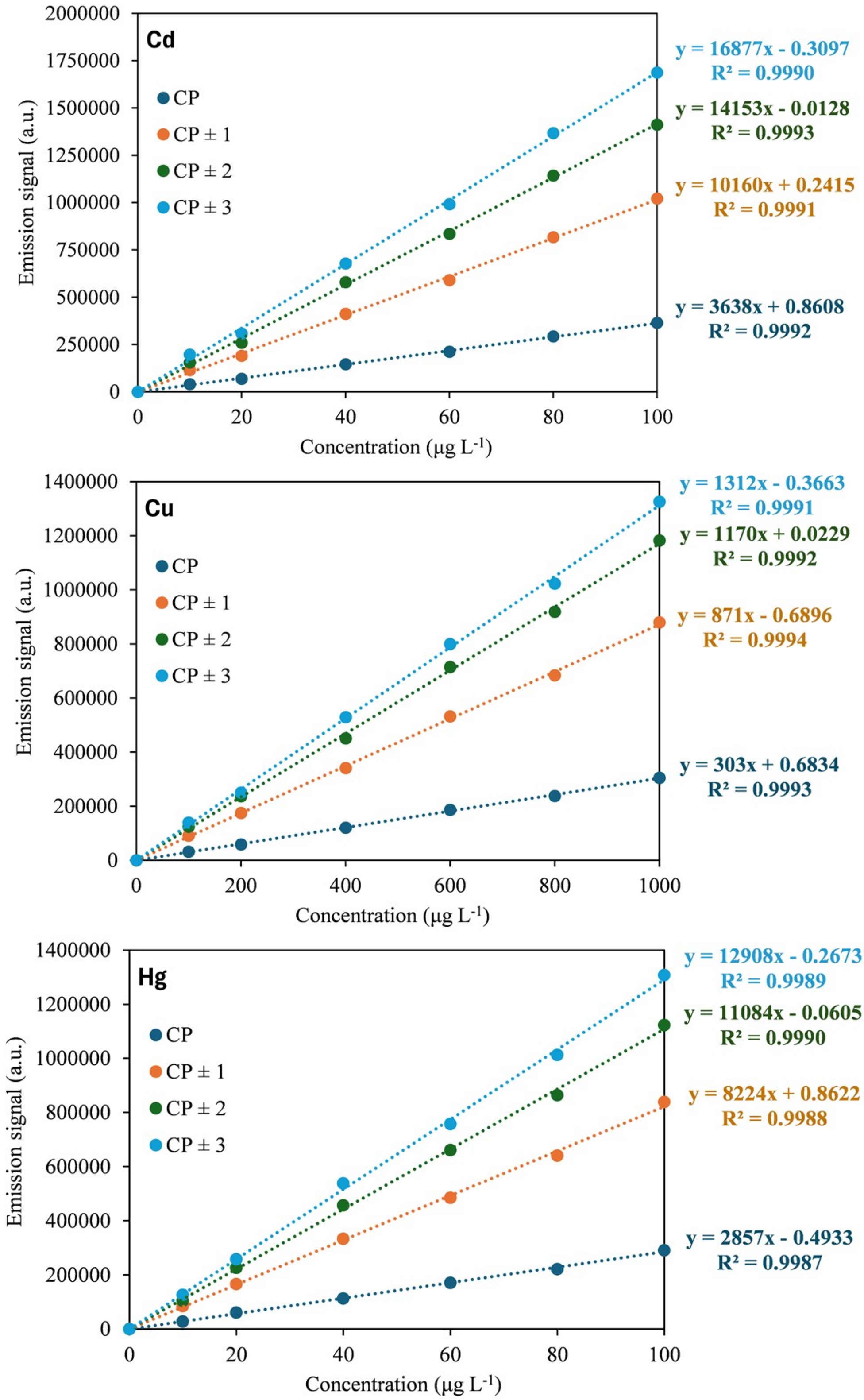


Fig. S1. Combustion efficiency (%) for 50 mg of GBW 10011 (Wheat) and TORT-3 (Lobster hepatopancreas) in flasks of 250, 500 and 1000 mL. Error bars correspond to relative standard deviation for n = 3 repeated measurements

2. Calibration curves obtained by SSETV-µCCP-OES method for different number of integrating pixels of the signal over the spectral line profile





Cd
CP
CP ± 1
CP ± 2
CP ± 3
y = 16877x - 0.3097
R² = 0.9990
y = 14153x - 0.0128
R² = 0.9993
y = 10160x + 0.2415
R² = 0.9991
y = 3638x + 0.8608
R² = 0.9992
Emission signal (a.u.)
Concentration (μg L-1)
Cu
y = 1312x - 0.3663
R² = 0.9991
y = 1170x + 0.0229
R² = 0.9992
y = 871x - 0.6896
R² = 0.9994
y = 303x + 0.6834
R² = 0.9993
Hg
y = 12908x - 0.2673
R² = 0.9989
y = 11084x - 0.0605
R² = 0.9990
y = 8224x + 0.8622
R² = 0.9988
y = 2857x - 0.4933
R² = 0.9987

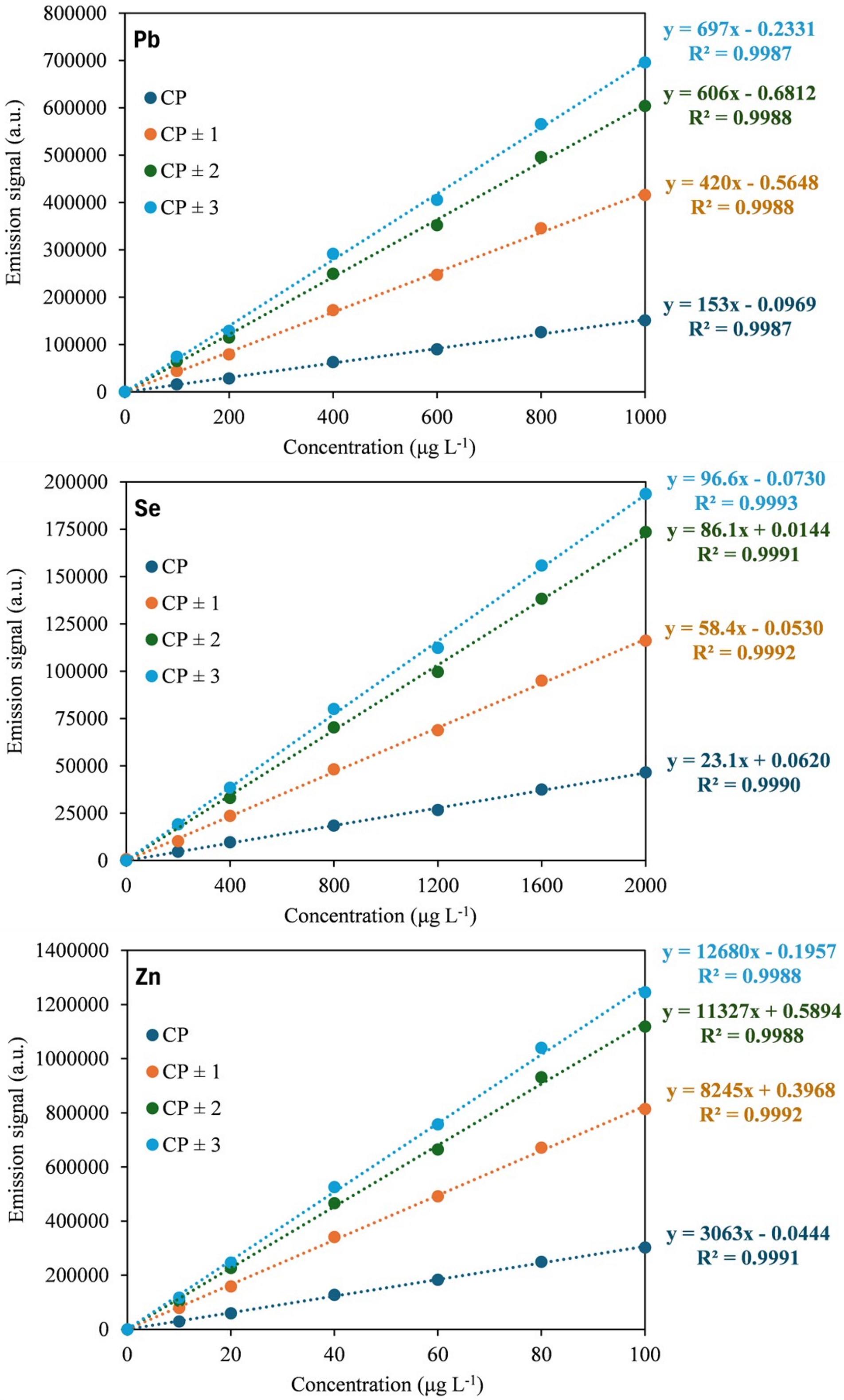


Fig. S2. Calibration curves obtained by SSETV-µCCP-OES method for different number of pixels used for signal integration over the spectral line profile (CP is the signal corresponding to the Central Pixel of the spectral line, ± 1; ± 2 and ± 3 corresponds to signals over the spectral line profile against CP)

3. Dependence of signal-to-background ratio, relative standard deviation of the background and LODs *versus* the number of integrating pixels of the signal over the spectral line profile

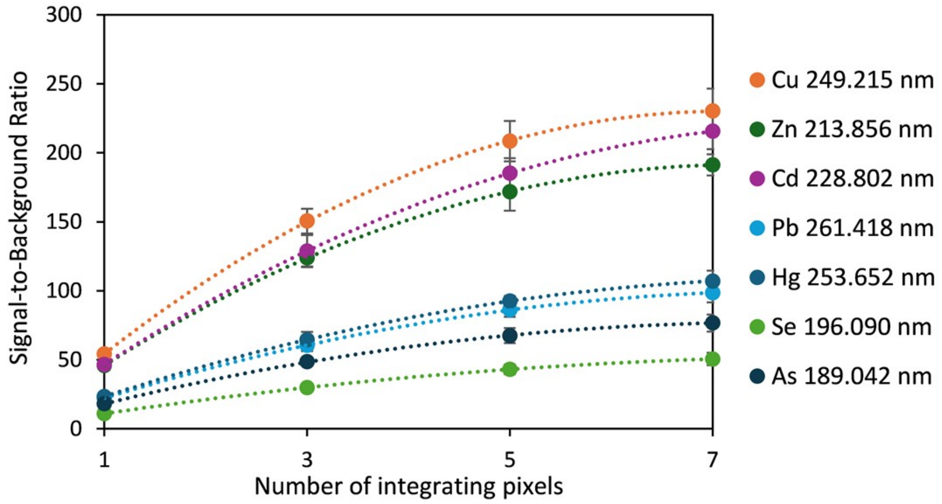


Fig. S3. Dependence of SBR in the SSETV-µCCP-OES method for different number of integrating pixels of the signal over the spectral profiles at the most sensitive lines of elements. Element concentrations (mg L^{-1}): 0.1 (Cd, Hg and Zn), 1 (Cu, Pb) and 2 (As, Se). Error bars correspond to standard deviation for n = 3 repeated measurements

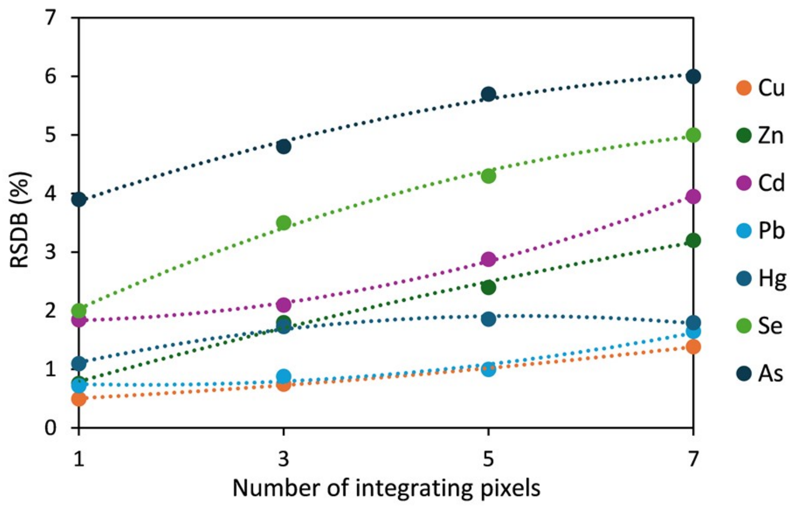


Fig. S4. Dependence of RSDB in the SSETV-µCCP-OES method for different number of integrating pixels of the signal over the spectral profiles of the most sensitive lines. Element concentrations (mg L^{-1}): 0.1 (Cd, Hg and Zn), 1 (Cu, Pb) and 2 (As, Se)

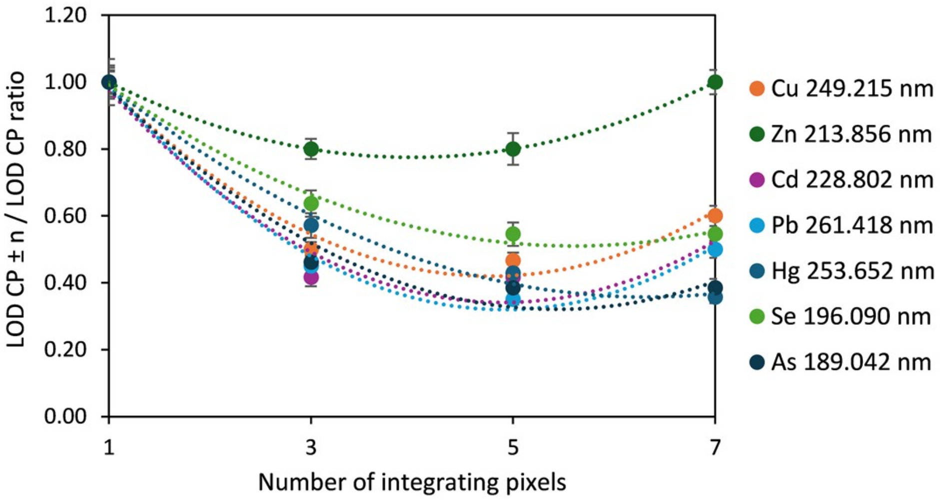
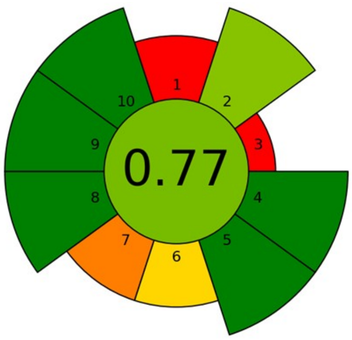
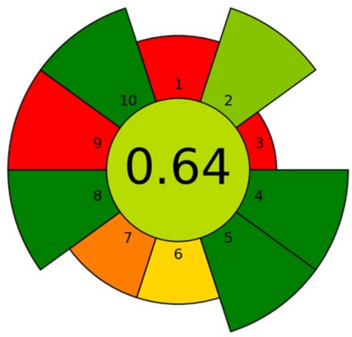


Fig. S5. Improvement of instrumental LODs in the SSETV-µCCP-OES method *versus* the number of integrating pixels of the signal over the spectral profile of the most sensitive lines. Error bars correspond to standard deviation for n = 3 repeated measurements.

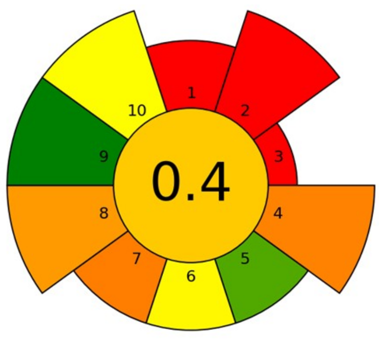
4. Colours of the (OFC)-SSETV-µCCP-OES method in comparison with traditional spectrometric methods



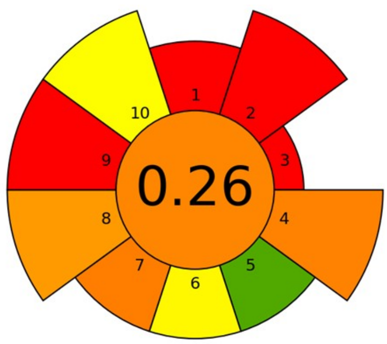
(OFC)-SSETV-µCCP-OES



(OFC)-ICP-OES



(HP-MAWD)-SSETV-µCCP-OES



(HP-MAWD)-ICP-OES

Fig. S6. The greenness of the (OFC)-SSETV-µCCP-OES method evaluated by the AGREEprep metric in comparison with (OFC)-ICP-OES, (HP-MAWD)-SSETV-µCCP-OES and (HP-MAWD)-ICP-OES methods. The assessment criteria, weight and inputs are presented in Table S2.

Table S2. Inputs used to assign the AGREEprep scores for the (OFC)-SSETV-µCCP-OES method in comparison with (OFC)-ICP-OES, (HP-MAWD)-SSETV-µCCP-OES and (HP-MAWD)-ICP-OES

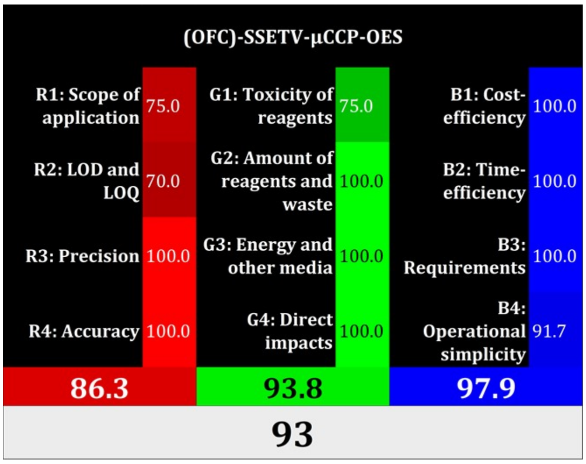
Assessment criteria	Method							
	(OFC)-SSETV-µCCP-OES		(OFC)-ICP-OES		(HP-MAWD)-SSETV-µCCP-OES		(HP-MAWD)-ICP-OES	
	Weight	Input	Weight	Input	Weight	Input	Weight	Input
1. Sample preparation placement	3	Ex-situ (in the lab)	3	Ex-situ (in the lab)	3	Ex-situ (in the lab)	3	Ex-situ (in the lab)
2. Hazardous materials	5	0.063 g concentrated HNO_3	5	0.063 g concentrated HNO_3	5	9 g HNO_3 + 1 g H_2O_2	5	9 g HNO_3 + 1 g H_2O_2
3. Sustainability, renewability, and reusability of materials	1	< 25% of reagents and materials are sustainable or renewable, but can only be used once	1	< 25% of reagents and materials are sustainable or renewable, but can only be used once	1	< 25% of reagents and materials are sustainable or renewable, but can only be used once	1	< 25% of reagents and materials are sustainable or renewable, but can only be used once
4. Waste	5	0.063 g concentrated HNO_3	5	0.063 g concentrated HNO_3	5	9 g HNO_3 + 1 g H_2O_2	5	9 g HNO_3 + 1 g H_2O_2
5. Size economy of the samples	5	0.050 g sample	5	0.050 g sample	3	0.3 g sample	3	0.3 g sample
6. Sample throughput	3	6 samples/h	3	6 samples/h	3	8 samples/h	3	8 samples/h
7. Integration and automation	3	2 steps in the sample preparation, manual system	3	2 steps in the sample preparation, manual system	3	2 steps in the sample preparation, manual system	3	2 steps in the sample preparation, manual system
8. Energy consumption	5	~ 0 Wh for OFC	5	~ 0 Wh for OFC	5	~ 150 Wh for sample digestion	5	~ 150 Wh for sample digestion
9. Post-sample preparation configuration for analysis	5	Simple, readily available detection: miniaturized instrumentation with low energy and Ar consumption	5	Advanced OES with high energy and Ar consumption: ICP-OES	5	Simple, readily available detection: miniaturized instrumentation with low energy and Ar consumption	5	Advanced OES with high energy and Ar consumption: ICP-OES
10. Operator's safety	5	No hazards or no exposure	5	No hazards or no exposure	5	2 Hazards	5	2 Hazards

Table S3. Evaluation tables by the RGB 12 algorithm for the (OFC)-SSETV-µCCP-OES method in comparison with (HP-MAWD)-ICP-OES, (HP-MAWD)-GFAAS and TDAAS

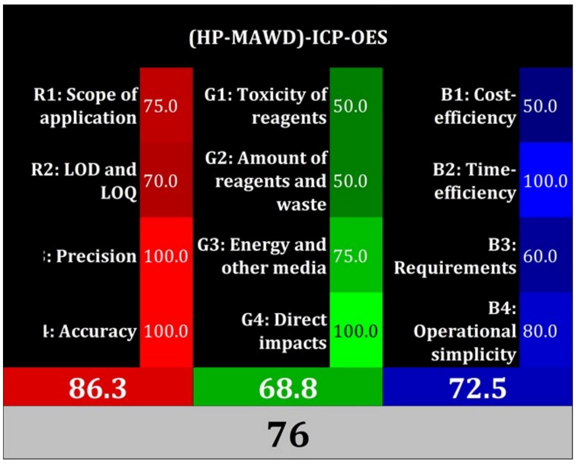
RED PRINCIPLES (analytical performance)			R1: Scope of application	R2: LOD and LOQ			R3: Precision			R4: Accuracy		
	Method number	Method name	0-100	LOD	LOQ	0-100	RSD% (repeatability)	RSD% (reproducibility)	0-100	Relative error (%)	Recovery (%)	0-100
	1	(OFC)-SSETV-µCCP-OES	75	0.01-1.2 mg kg^{-1}	0.03-4.0 mg kg^{-1}	70	4.6-14.5	-	100	-	92-113	100
	2	(HP-MAWD)-ICP-OES	75	0.04-0.85 mg kg^{-1}	0.13-2.80 mg kg^{-1}	70	6.7-13.7	-	100	-	91-111	100
	3	(HP-MAWD)-GFAAS	75	0.006-0.03 mg kg^{-1}	0.020-0.10 mg kg^{-1}	100	4.1-11.8	-	100	-	85-107	100
	4	TDAAS	50	0.004 mg kg^{-1}	0.013 mg kg^{-1}	100	24	-	80	-	92-108	100

GREEN PRINCIPLES (green chemistry)			G1: Toxicity of reagents (impact and biodegradation)		G2: Amount of reagents and waste			G3: Consumption of energy and other media	G4: Direct impacts (safety, use of animals and GMOs)			
	Method number	Method name	Total number of pictograms	0-100	Reagent consumption	Waste production	0-100	1-100	Occupational hazards	Safety of users (0-100)	Use of animals (0 if no, 1 if yes)	Use of GMO (0 if no, 1 if yes)
	1	(OFC)-SSETV-µCCP-OES	3	75	0.063	0.063	100	100	-	100	0	0
	2	(HP-MAWD)-ICP-OES	9	50	20	20	50	75	-	100	0	0
	3	(HP-MAWD)-GFAAS	3	75	10	10	60	90	-	100	0	0
	4	TDAAS	0	100	0	0	100	90	-	100	0	0

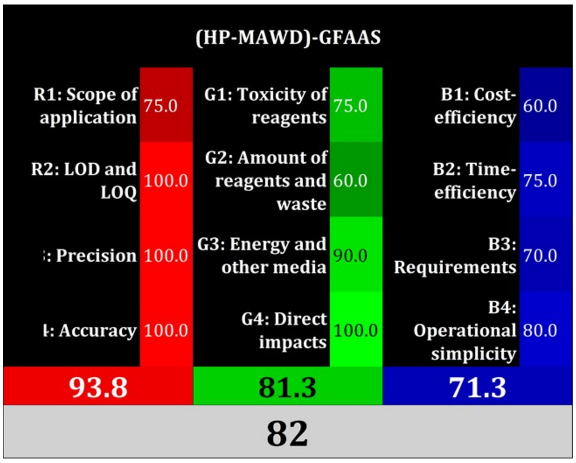
BLUE PRINCIPLES (practical side)			B1: Cost-efficiency		B2: Time-efficiency		B3: Requirements			B4: Operational simplicity		
	Method number	Method name	Total cost	0-100	Speed of analysis	0-100	Sample consumption	Sample consumption (0-100)	Other needs: advanced instruments, skills, facilities (0-100)	Miniaturization (0-100)	Integration and automatization (0-100)	Portability (0-100)
	1	(OFC)-SSETV-µCCP-OES	Very low	100	High	100	0.05 g	100	100	100	75	100
	2	(HP-MAWD)-ICP-OES	High	50	High	100	0.3 g	70	50	70	100	70
	3	(HP-MAWD)-GFAAS	Medium	60	Medium	75	0.3 g	70	70	70	100	70
	4	TDAAS	Medium	75	Medium	60	0.2 g	80	100	80	100	70



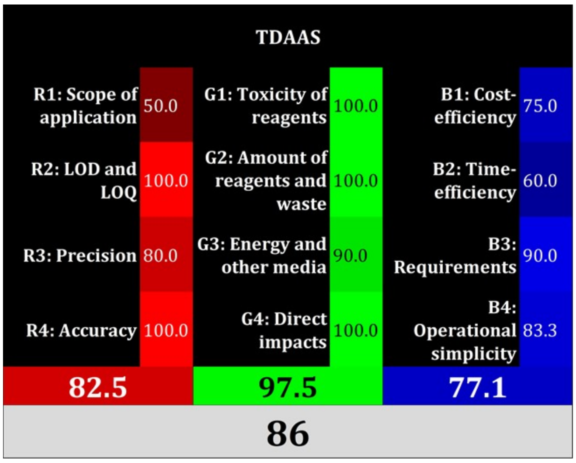
(OFC)-SSETV-µCCP-OES



(HP-MAWD)-ICP-OES



(HP-MAWD)-GFAAS



TDAAS

Fig. S7. The colours of the (OFC)-SSETV-µCCP-OES method evaluated by the RGB 12 algorithm in comparison with (HP-MAWD)-ICP-OES, (HP-MAWD)-GFAAS and TDAAS methods. The considered inputs are illustrated in Table S3.