



Determination of 14 Elements in Botanical Samples by Simultaneous Inductively Coupled Plasma Atomic Emission Spectrometry Using Standard Reference Material as Multielement Standard

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Bestimmung von 14 Elementen in botanischen Proben durch simultane ICP-Atomemissionsspektrometrie mit Hilfe von Standard-Referenzmaterial als Multielement-Standard

Zusammenfassung. Eine Methode wird beschrieben, bei der NBS-Standard-Referenzmaterial SRM 1571 (orchard leaves) als Multielement-Standard zur Bestimmung von 14 Elementen (einschließlich Haupt-, Neben- und Spurenelemente) in 2 NBS- und 3 BCR-Standardmaterialien mit Hilfe der simultanen ICP-Atomemissionsspektrometrie verwendet wurde. Sehr gute Übereinstimmung der Ergebnisse mit den zertifizierten Werten wurde für 11 Elemente erhalten. Niedrige Werte für Al, Ti und Fe waren auf unvollständigen Aufschluß (unzulängliche Extraktion) bei der Veraschung unter Druck mit Salpetersäure zurückzuführen. Diese 3 Werte können jedoch als salpetersäurelöslicher Anteil angesehen werden. Die Interelement-Störungen durch die Hauptbestandteile (Ca, Mg, Al, Mn, Fe) werden diskutiert und korrigiert. Die paarweisen Aufschlußlösungen von NBS SRM 1571 wurden auch als Standards zur Qualitätskontrolle benutzt. Das Verfahren ergibt genaue und zuverlässige Ergebnisse.

Summary. This paper describes the method, in which NBS (National Bureau of Standards) SRM 1571 (Standard Reference Material, Orchard Leaves) was used as multielement standard to determine 14 elements including major, minor and trace elements in 2 NBS and 3 BCR (Community Bureau of Reference of the European Communities) botanical Standard Reference Materials by simultaneous ICP-AES (Inductively Coupled Plasma Atomic Emission Spectroscopy). Very good agreement between the results and the certified values or information data for 11 elements was found. The low values for Al, Ti and Fe were due to incomplete digestion (extraction inefficiency) by pressure ashing with nitric acid. But these three values can be regarded as the nitric acid soluble portion. The interelement interferences caused by the major components such as Ca, Mg, Al, Mn and Fe were discussed and corrected. The paired digestion solutions of NBS SRM 1571 were also used as quality control standards. Accurate and reliable analytical results can be obtained by this method.

Introduction

The studies involved in different fields about the roles and effects of trace elements in biological systems are receiving worldwide attention. To achieve a better understanding about the functions of trace elements, accurate and reliable analytical data are indispensable. With the application of various new analytical techniques and deepgoing studies in trace element analysis, it will often be found that there are discrepant analytical data for the same sample by different techniques and laboratories. In order to obtain accurate, reliable and useful analytical data, the so-called quality control [1, 2] is becoming increasingly more and more important. As far as the analytical method is concerned, the Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) has many advantages for the simultaneous multielement analysis of a variety of biological samples. In recent publications [3, 4] the quality control procedures in ICP-AES have been emphasized. The use of certified standard reference materials is one of the important steps in the quality control procedures. Many authors have elucidated the importance of standard reference materials [5–7]. The performance of laboratories using a standard reference material was better than that of those who did not use standard reference material [8, 9]. In this paper the use of SRM 1571 (Orchard Leaves) is reported, which is the most commonly used quality control standard, as multielement calibration standard. The use of “synthetic” standards, where known amounts of elements are prepared in the individual laboratory has been the predominant method used in ICP for many years. But this technique has a number of limitations including the preparation, dilution and mixing of a large number of standard solutions with subsequent errors like contaminations caused by the impurities in the element compounds added as well as the problem of stability during storage over a long time. Chaplin and Dixon [10] found that synthetic reference solutions provided unsuitable recoveries and necessarily relied, for instrumental calibration, upon availability of previously characterized standard reference materials. Scott and Strasheim [11] have determined six elements in six botanical samples. They were able to employ a synthetic reference solution for calibration, but found it necessary to match approximately the matrix composition of the samples to obtain satisfactory accuracy of determination. McQuaker et al. [12] found that by confining each standard solution to only one concentration level any cross contamination due to impurities in the stock solution was insignificant. This

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compared well with calibrations where a range of concentration levels has been grouped together in individual standards making contamination correction necessary [13, 14]. They also used selected reference materials to assess calibration performance in terms of analytical precision and accuracy. The use of standard reference materials as irradiation standards in Neutron Activation Analysis (NAA) was first introduced by Morrison et al. [15] in 1968. Now this method has been widely used in NAA [16]. These standard reference materials, which are normally used to check various multielement analytical methods, provide convenient solid multielement standards for irradiation involving 30 to 40 elements. They allow simple and reproducible drying, long stability during storage and eliminate most of preparation work in NAA. Standard reference materials were also used as solid standard providing calibration points in solid-sampling electrothermal atomic absorption spectrometry [17–19]. Alexander and McAnulty [20] used NBS standard reference materials for calibration in the multielement analysis of plant tissues by arc and spark emission spectroscopy. Good accuracy was achieved by this method. Brätter et al. [21] reported recently the use of NBS SRM 1577 (Bovine Liver), 1571 (Orchard Leaves) and Bowen's Kale as calibration standards in the multielement analysis of biological material by simultaneous ICP. The digested solutions of standard reference materials were stepwise diluted to set up the calibration curve. They reported that errors in the background correction became negligible if a similar matrix was present between the sample and standards. They did not mention the interelement interference correction. But from our preliminary experiments satisfactory results will not be obtained unless the interelement interference correction and background correction are made. The reason is the big difference in the matrix composition among plant tissue samples. In this paper we also describe the routine use of NBS SRM 1571 to provide calibration points for the determination of 14 elements in 5 botanical standard reference materials by ICP and to be incorporated into this method as the recalibration solutions. Interelement interference and background correction were made because of the concomitant stray radiation and direct or wing spectral interferences. In addition, the digested SRM 1571 was used as quality control solution during the running of measurement to correct all bias from the certified values. Two NBS botanical SRM's and three botanical SRM's of the European Community Bureau of Reference (BCR) were used for verifying this analytical method.

Experimental

Instrumentation

The following ICP instrumentation was employed: Inductively Coupled Plasma Emission Spectrometer: model JY-38P (Instruments S.A.), as previously described [22] and JY-48 (Instruments S.A.). They are mounted at right angle using the same ICP device. The 14 mounted analytical wavelengths are shown in Table 1. Background correction was performed on those channels where wavelength scanning had shown that it was necessary. By means of a primary slit translation system the entire emission spectrum in the exit focal plane was shifted. The wavelengths and the positions at which background correction was made are also shown in Table 1. The power input to the plasma was 0.9 kW. The coolant argon gas

Table 1. ICP wavelengths and background correction positions

Element	Wavelength, nm	Background correction position	
		low side of analytical line	high side of analytical line
Ca	II 393.366	393.280	393.426
Mg	II 279.553	279.467	279.613
Mn	II 257.610	257.524	257.670
Fe	II 259.940	— ^a	260.000
Al	I 396.152	396.066	396.212
Cr	II 267.716	267.630	267.776
V	II 311.071	—	311.131
Be	II 313.107	—	313.135
Ti	II 334.941	334.855	—
Ni	II 231.604	231.518	231.664
Cu	I 324.754	324.668	324.814
Pb	II 220.353	220.267	—
Zn	I 213.856	213.770	213.916
Cd	II 226.502	—	226.562

^a Dash indicates no background correction

flow rate was 14.0 l/min. The carrier argon gas mixed with 6.5% hydrogen was measured and controlled by a mass flow meter and controller. The pressure of carrier gas was 2.0 bar. The observation height was 12 mm above rf load coil. The preintegration time and integration time were 30 s and 10 s, respectively, due to the application of a sample changer.

Sample Materials

For testing the method the following standard reference materials have been used: NBS SRM 1571 (Orchard Leaves), 1570 (Spinach), and 1573 (Tomato Leaves); BCR SRMs No. 60 and 61, these are two plant materials of aquatic origin (water pest and water moss); BCR SRM No. 62 (Olive Leaves).

Sample Preparation Method

All weights mentioned in this paper were dry weight. Standard reference materials as well as samples were dried under the conditions suggested by NBS, i.e., at 85°C for 2 h. The dry weight of sample divided by the wet weight was the drying factor. About 0.5 g of samples were weighed into quartz digestion tubes. 4 ml of sub-boiling distilled nitric acid were added to each tube. The tubes were put into an autoclave which was described earlier [23] and then placed in a temperature-controlled heating block in 140°C for 6 h. After being cooled, the digested samples were rinsed into 25 ml volumetric flasks and diluted to volume with deionized water. When the standard reference materials of BCR were digested, 0.1 g of samples added to 1 ml of nitric acid was digested in a smaller autoclave and diluted to 10 ml. In order to extend the calibration range a certain amount of some element (see the following sections) were added to the digested SRM 1571 solution before diluting to 25 ml. In most botanical samples there was an insoluble fraction (silica) left after wet decomposition. All solutions were allowed to settle. The clear rest was transferred into a quartz ampoul and stored until measurement.

Table 2. Calibration scheme

Element	Dilution factor und content level µg/g						
	1 × ^a	1 × (CAL 002)	2 ×	5 ×	10 × (CAL 001)	20 ×	50 ×
Ca		20,900	10,450	4,180	2,090	1,045	418
Mg		6,200	3,100	1,240	620	310	124
Mn	182	91	45.5	18.2	9.1	4.55	1.82
Fe		270	135	54	27	13.5	5.4
Al	524	262	131	52.4	26.2	13.1	5.3
Cr ^b		6.6			0.66		
V		3.04	1.52	0.608	0.304	0.152	
Be		0.256	0.128	0.0512	0.0256	0.013	
Ti	9.1	4.6	2.25	0.91	0.45	0.23	
Ni	12.6	6.3	3.15	1.26	0.63	0.315	
Cu	24	12	6	2.4	1.2	0.6	
Zn	50	25	12.5	5	2.5	1.25	
Pb		44	22	8.8	4.4	2.2	
Cd		5.1	2.55	1.02	0.15	0.255	

^a See text^b In view of the severe interelement interference the calibration set for Cr was prepared by the progressive dilution of 10 µg/ml Cr solution in a six points calibration: 10, 20, 50, 100, 200 and 500 ng/ml. This corresponded to 0.5, 1, 2.5, 5, 10 and 25 µg/g, respectively, if 0.5 g of dry sample was weighed, digested and diluted to 25 ml

Standard and Calibration Curve

When SRM 1571 (Orchard Leaves) was used as calibration standard 0.5219 g of Orchard Leaves (drying factor was 0.958) was weighed in duplicate and digested. Referring to the elemental content data of some botanical NBS SRM, some elemental contents in Orchard Leaves such as Ni, Cd and so on are too low to provide wide analytical range. Therefore, 2.5 µg Cd, 2.5 µg Ni, 2.0 µg Cr, 1.2 µg V and 0.12 µg Be were added to one of them. The certified Ni content is 1.3 µg/g. So the total amount of Ni in 0.5219 g (equals 0.5000 g dry weight) is 3.15 µg, which corresponds to 6.3 µg/g in Orchard Leaves. The same method was used for other spiked elements. The values are shown in Table 2. 45.5 µg of Mn, 12.5 µg of Zn, 131 µg of Al, 6 µg of Cu, 5.7 µg of Ni and 2.3 µg of Ti were added to another digested SRM 1571 solution to extend further the calibration curve. Then both were diluted to 25 ml. The first solution was called CAL002 and used as stock standard solution. The CAL 002 was diluted progressively to get the complete set of calibration standard over five to six concentration points, i.e., stock standard solution 1 ×, 2 ×, 5 ×, 10 ×, 20 ×, and 50 ×. The numbers were dilution factors. The solution with dilution factor 10 was called CAL 001. The whole calibration scheme is shown in Table 2.

Data Acquisition and Calculation

The whole program of JY-48 used in this work for data acquisition is illustrated in Fig. 1 in the form of a flow chart. More detailed information about programming is given in the specification of the JY-48 ICP spectrometer. All measured intensities as well as its corresponding elemental content values in Table 2 were fitted to a linear regression curve or a second order polynomial with 5–7 data points. The calibration curve was stored in the regression curve file of computer software. In the later routine analysis, the CAL 001 and 002 were used as recalibration standards to validate the calibration curve. This means that the intensities of CAL 001

and CAL 002 were compared by the computer with the stored one of the original curve to obtain coefficients which normalized the intensities of subsequent samples before the calibration coefficients were applied. At the same time, in order to ensure the validity of the final results CAL 001 and 002 were used as quality control standards to compensate the measured values which departed from the certified values. They were measured after each group of ten samples. The importance of measuring paired solutions of known concentrations for routine quality control has been emphasized by King [24], Moselhy and Vijan [25]. The final result was calculated from the formula:

$$R(\mu\text{g/g}) = R_{\text{mes.}} \cdot f_{\text{dil.}} \cdot \frac{f_{\text{std.d.}}}{f_{\text{sam.d.}}} \cdot \frac{W_{\text{std.}}}{W_{\text{sam.}}} \cdot \frac{V_{\text{sam.}}}{V_{\text{std.}}} \cdot \frac{C}{\left(\frac{C_b + C_a}{2}\right)}$$

in which $R_{\text{mes.}}$ is the measured result, $f_{\text{dil.}}$ is the dilution factor, which is defined as dilution steps from digested sample ($f_{\text{dil.}} = 1$ if no further dilution). $f_{\text{std.d.}}$ and $f_{\text{sam.d.}}$ are the drying factors of standard and sample, respectively. $W_{\text{std.}}$ and $W_{\text{sam.}}$ are the weight of standard and sample, respectively. $V_{\text{std.}}$ and $V_{\text{sam.}}$ are the volume of digested standard and sample, respectively, C is the certified value of one quality control standard. C_b and C_a are the measured values of that quality control standard before and after the set of samples are measured.

Results and Discussion

Multielement Calibration Standard

In selecting a multielement calibration standard for the analysis of botanical samples a botanical standard reference material is preferred. NBS SRM 1571 (Orchard Leaves) is the most commonly used quality control material. The certificate of SRM 1571 [26] provides 11 elemental content values

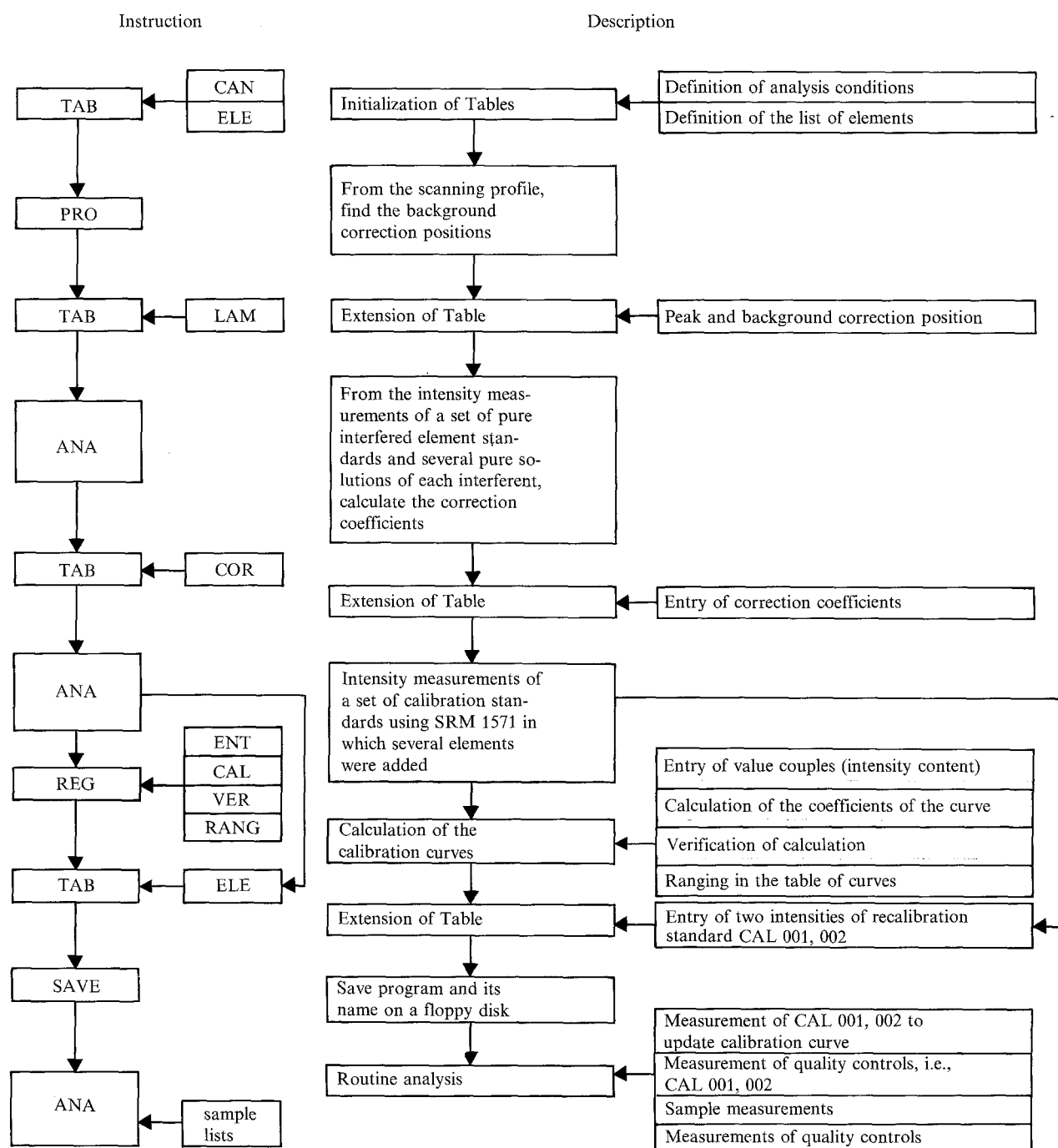


Fig. 1. Flow chart of program of JY-48

among 14 elements determined in this work. Because the success of the SRM multielement standard approach to ICP depends on the validity of the elemental content values, the uncertified elemental contents such as Al, V, Ti were determined firstly by ICP using the sequential JY-38 spectrometer. In Table 3 the results including Fe and Be were compared with the literature data. The content of Al was comparable with other values [13, 27] by the ICP method. The Be value was different from the certified value as previously reported [28]. The Fe and V values were in excellent agreement with literature data. The Fe, Al and Ti values may represent the nitric acid soluble portions. The data obtained in this work together with other certified elemental contents will be entered into the calibration curve.

Table 3. Elemental contents ($\mu\text{g/g}$) in NBS SRM 1571

Element	This work	NBS	Gladney [31]	Other source
Al	262 ± 11^a		300 ± 100	286 [13]
Be	0.019 ± 0.002	0.027 ± 0.01		0.015 ± 0.002 [28]
Fe	273 ± 15	270^b		262 [13] 273 ± 6 [10] 267 ± 6 [29]
Ti	4.5 ± 0.4		20 ± 20	
V	0.64 ± 0.10		0.6 ± 0.15	0.54 [30] 0.61 [16]

^a The mean value $\pm$ the 95% confidence interval

^b $270 \mu\text{g/g}$ is the nitric-perchloric acid soluble portion

Table 4. Interelement interference correction

Analyte	Wavelength nm	Interfering element	Correction coefficient
Al	396.152	Ca	1.1×10^{-3a}
Pb	220.353	Al	2.1×10^{-3}
		Mg	0.4×10^{-4}
Cr	267.716	Mg	0.23×10^{-3}
Ni	231.604	Mg	0.15×10^{-3}
Cd	226.502	Mg	0.1×10^{-4}
		Fe	0.8×10^{-4}
V	311.071	Mn	0.31×10^{-3}
Zn	213.856	Ni	4.2×10^{-3}

^a Correlations were made on a content equivalent basis, i.e. $\mu\text{g/g}/\mu\text{g/g}$

Interelement Interference Correction

In preliminary experiments of this work we failed to determine some trace elements such as Cr, V, Cd and Pb when the interelement interference correction was not performed. Later it was found that the major matrix constituents of botanical samples such as Ca, Mg, Al, Fe and Mn influenced the accuracy of the determination. The direct spectral overlap or the overlap caused by the wing of the interference line and stray light effects were the major sources of interelement interference effects. The analyte channel was calibrated with its corresponding pure element solution and at the same time the apparent analyte units were obtained by measuring several pure solutions of each of the interferents. In this work the concentration of the interfering elements was referred to the NBS botanical standards. The correction was made on a content equivalent basis. A linear relationship between the apparent analyte/interferent data was assumed. Dahlquist and Knoll [13] pointed out that the interferent response functions were not linear. They employed coefficients of a second-order polynomial regression of the experimental data pairs for spectral interference corrections. We found that the difference was negligible if the concentration of interfering element was in the range in which the coefficients of correction were obtained. The obtained correction coefficients are given in Table 4. It was necessary to point out that these correction coefficients were unique to our instruments and ICP operating parameters. In the case of Cr the low analytical result was obtained if the calibration curve for Cr was established by progressive dilution of SRM 1571 spiked with Cr as given in Table 2. It was found that the slope of this curve was higher than that of pure Cr solution. The reasons were the big influence of Mg and low content of Cr in calibration standard. In the case of other elements either the analyte contents in calibrations standard such as Pb, Al were high or the interfering element contents such as Fe, Mn were low. Such interelement influence on the calibration curve was not obvious and this small influence could be compensated by the quality control standard. Therefore the calibration curve of Cr was replaced by pure Cr solution as given in the footnote of Table 2.

Comparison of Accuracy with Certified and Information Values

Table 5 is the summary of results obtained in this work and comparison of accuracy with the certified values of 14 elements of 2 botanical standard reference materials, i.e.,

Table 5. Elemental concentration in two NBS botanical standard reference materials

Element	Sample name		NBS SRM 1573 (tomato leaves)				
	Content and confidence interval		NBS SRM 1570 (spinach)				
	95% $\mu\text{g/g}$		This work	NBS [32]	Gladney [31]	Munter [27]	Wolnik [3]
Al	620 $\pm$ 36			870 $\pm$ 50	730 $\pm$ 220	609 $\pm$ 16	
Be	0.016 $\pm$ 0.006				< 0.06		
Ca	14,400 $\pm$ 350			13,500 $\pm$ 300	15,200 $\pm$ 800	12,900 $\pm$ 440	12,900 $\pm$ 300
Cd	1.4 $\pm$ 0.2			(1.5) ^a	1.6 $\pm$ 0.3	1.65 $\pm$ 0.33	1.5 $\pm$ 0.1
Cr	4.2 $\pm$ 0.7			4.6 $\pm$ 0.3	4.9 $\pm$ 0.6	3.7 $\pm$ 1.2	4.3 $\pm$ 0.7
Cu	10.7 $\pm$ 0.5			12 $\pm$ 2	11.5 $\pm$ 1.4	10.5 $\pm$ 0.3	11.8 $\pm$ 0.2
Fe	508 $\pm$ 14			550 $\pm$ 20	512 $\pm$ 38	576 $\pm$ 18	525 $\pm$ 11
Mg	8,800 $\pm$ 270				8,600 $\pm$ 1,200	8,500 $\pm$ 120	8,600 $\pm$ 230
Mn	167 $\pm$ 5			165 $\pm$ 6	149 $\pm$ 27	176 $\pm$ 2	165 $\pm$ 3
Ni	5.5 $\pm$ 0.5			(6)	5.3	5.7 $\pm$ 0.3	5.5 $\pm$ 0.3
Pb	2.2 $\pm$ 0.6			1.2 $\pm$ 0.2	1.2 $\pm$ 0.1		
Ti	8.9 $\pm$ 1.4				16.5		
V	1.5 $\pm$ 0.2				1.3 $\pm$ 0.3		[16]
Zn	47.0 $\pm$ 2.5			50 $\pm$ 2	57 $\pm$ 12	51.6 $\pm$ 4	49.8 $\pm$ 1.3
							1.2 $\pm$ 0.1
							1.5 $\pm$ 0.2
							59.5 $\pm$ 2.2
							62 $\pm$ 6
							6.3 $\pm$ 0.5
							6.1
							68
							[16]
							1.3
							1.27 $\pm$ 0.03
							58.3 $\pm$ 3.3

^a The bracketed values are information values

Table 6. Elemental concentration in three BCR botanical reference materials [36]

Content and confidence interval 95 % $\mu\text{g/g}$	Sample name					
	BCR RM No. 60 (water pest)		BCR RM No. 61 (water moss)		BCR RM No. 62 (olive leaves)	
	This work	BCR [36]	This work	BCR	This work	BCR
Al	$1,840 \pm 120$	(6,140) ^a 1,800 ^b	$4,300 \pm 190$	(17,150) $4,700 \pm 340^b$	435 ± 25	(265) 484 ± 10^b
Be	0.086 ± 0.007		0.196 ± 0.015		< 0.015	
Ca	$30,540 \pm 750$	(30,950)	$16,190 \pm 500$	(16,940)	$19,080 \pm 380$	(17,520)
Cd	2.3 ± 0.2	2.20 ± 0.10	1.70 ± 0.18	1.07 ± 0.08	< 0.2	0.10 ± 0.02
Cr	27.2 ± 2.1	29.6 ± 0.5^b	503 ± 38	547 ± 10^b	1.5 ± 0.2	1.7 ± 0.1^b
Cu	49.1 ± 3.4	51.2 ± 1.9	683 ± 14	720 ± 31	45.5 ± 2.8	46.6 ± 1.8
Fe	$2,100 \pm 120$	(2,380)	$8,440 \pm 400$	(9,300)	317 ± 8	(280)
Mg	$6,050 \pm 60$	(6,030)	$3,920 \pm 100$	(3,920)	$1,170 \pm 16$	(1,206)
Mn	$1,860 \pm 60$	$1,760 \pm 50$	$3,996 \pm 97$	$3,771 \pm 80$	59.8 ± 1.4	57.0 ± 2.4
Ni	40.5 ± 2.0	(40)	463 ± 19	(420)	1.5 ± 0.1	(8)
Pb	67.4 ± 2.9	63.8 ± 3.2	62.6 ± 1.5	64.4 ± 3.5	25.8 ± 1.2	25.0 ± 1.5
Ti	33.0 ± 2.4	(240)	65.3 ± 4.7	(780)	4.7 ± 0.3	(240)
V	5.2 ± 0.6	(6)	12.7 ± 1.0	(6)	0.93 ± 0.11	(1)
Zn	322 ± 10	313 ± 8	583 ± 17	566 ± 13	17.2 ± 0.6	16.0 ± 0.7

^a The bracketed values are information values

^b The results were obtained by graphite furnace atomic absorption spectrometry using the same digestion method in this laboratory

NBS 1570 and 1573. Other literature information values are also given. Table 6 compares the certified values of 5 elements for the 3 botanical standard reference materials of the Community Bureau of Reference, i.e., BCR SRM No. 60, 61 and 62. Reference to Tables 5 and 6 indicates that in spite of the high degree of variability in element distribution in the different kinds of plant materials the agreement between our data and certified as well as information values is excellent with a few exceptions. Some low values appear. These include Al and Ti. Fe is also a little lower than the certified data. Some authors [26, 29, 34] pointed out that the low ICP-AES results for Fe, Al and Ti using the mixed acid digestion method ($\text{HNO}_3/\text{HClO}_4/\text{HF}$) were due to the incomplete dissolution or silica occlusion. It is possible that these elements are present in several forms of binding associated with the silicate matrix. But the results for Al, Fe and Ti might be regarded as the nitric acid soluble portion. The Be value in BCR No. 61 is nearly the same as that previously reported [28]. Al and Cr contents in three BCR samples were also measured independently by GFAAS using the same digestion method. The results were in good agreement with that obtained by ICP using NBS SRM 1571 as calibration standard. From the Tables 5 and 6 it can be seen that the pressure digestion method with nitric acid is adequate for most of the elements in the botanical samples.

Conclusion

It is obvious from the data in Tables 5 and 6 that ICP using SRM as multielement standard is capable of providing accurate results for a variety of botanical samples. The biological SRM would be best used to analyse biological materials since the match between standards and samples would be closest. From the results shown in Tables 5 and 6, however, this close match actually is not essential since the good agreement with certified values can be obtained for most elements even if the contents of some elements in different

types of botanical samples may differ in one to two orders of magnitude. It is possible that this method can be extended to other matrix samples if the interelement correction coefficients are obtained, background interferences are performed and the calibration curves are proper. With the appearance of new standard reference materials and the decrease of the price owing to international cooperation in the field of reference materials [37] this kind of method will certainly develop to gain accurate and reliable analytical data.

References

1. Taylor JK (1981) *Anal Chem* 53:1588 A
2. Horwitz W (1982) *Anal Chem* 54:67 A
3. Wolnik KA, Fricke FL, Kuennen RW (1982) *ICP Inf Newsl* 8:4
4. Botto RI (1982) *ICP Inf Newsl* 8:1
5. Stoeppler M, Valenta P, Nürnberg HW (1979) *Fresenius Z Anal Chem* 297:22
6. Cali JP (1979) *Fresenius Z Anal Chem* 297:1
7. Uriano GA, Gravatt CC (1977) *CRC Crit Rev Anal Chem* 6:361
8. Koh TS, Benson TH, Judson GJ (1980) *Assoc Offic Anal Chemists* 63:809
9. Michel R, Freisen-Broda G, Hombach V, Tauchert M (1982) Systematic errors and quality assurance in the trace element analysis of human serum, presented at 2nd International Workshop, Trace Element Analytical Chemistry in Medicine and Biology, April 21–24th 1982, Neuherberg, FRG
10. Chaplin MH, Dixon AR (1974) *Appl Spectrosc* 28:5
11. Scott RH, Strasheim A (1975) *Anal Chim Acta* 76:71
12. McQuaker NR, Kluckner PD, Chang GN (1979) *Anal Chem* 51:888
13. Dahlquist RL, Knoll JW (1978) *Appl Spectrosc* 32:1
14. Church SE (1978) Paper 387, presented at the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, Cleveland, Ohio, USA
15. Peterson SF, Travesi A, Morrison GH (1968) *Modern trends in activation analysis*, vol 1. Gaithersburg, Md
16. Nadkarni RA, Morrison GH (1978) *J Radional Chem* 43:347
17. Nichols JA, Jones RD, Woodriff R (1978) *Anal Chem* 50:2071
18. Langmyhr FJ, Orre S (1980) *Anal Chim Acta* 118:307
19. Takada K, Hirokawa K (1982) *Fresenius Z Anal Chem* 312:109

20. Alexander GV, McAnulty LT (1981) *J Plant Nutr* 3:51
21. Braetter P, Berthold KP, Gardiner PE (1982) Paper presented at 1982 Winter Conference on Plasma Spectrochemistry, January 4–9, 1982, Orlando, Florida, USA
22. Schramel P, Xu LQ (1982) *ICP Inf Newsl* 7:429
23. Schramel P, Wolf A, Seif R, Klose BJ (1980) *Fresenius Z Anal Chem* 302:62
24. King DE (1976) US-NBS special publication, 402, *Proc 7th Material Research Symposium* 1:141
25. Moselhy MM, Vijan PN (1981) *Anal Chim Acta* 130:157
26. National Bureau of Standards (US), certificate of analysis for Standard Reference Material 1571, orchard leaves, NBS, Washington, Revision 31, Aug 1977
27. Munter RC, Grande RA, Ahn PC (1979) *ICP Inf Newsl* 5:368
28. Schramel P, Xu LQ (1982) *Anal Chem* 54:1333
29. McQuacker NR, Brown DF, Kluckner PD (1979) *Anal Chem* 51:1082
30. Kosta L (1980) Technical Reports Series No 197. 317. International Atomic Energy, Vienna
31. Glaney ES (1980) *Anal Chim Acta* 118:385
32. National Bureau of Standards (US), certificate of analysis for Standard Reference Material 1570, spinach, NBS, Washington, Sep 1976
33. National Bureau of Standards (US), certificate of analysis for Standard Reference Material 1573, tomato leaves, NBS, Washington, Oct 1976
34. McLaren OW, Berman SS, Boyko VJ, Russell DS (1981) *Anal Chem* 53:1802
35. Guzzi G, Pietra R, Sabbioni E (1976) *J Radional Chem* 34:35
36. Muntau H, Griepink B, Colinet E (1983) *Fresenius Z Anal Chem* (in print)
37. Neider RJA (1979) *Fresenius Z Anal Chem* 297:4

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