

A SEPARATION SCHEME FOR THE DETERMINATION OF TRACE ELEMENTS IN BIOLOGICAL MATERIALS BY NEUTRON ACTIVATION ANALYSIS

P. LIEVENS*, R. CORNELIS** and J. HOSTE

Institute for Nuclear Sciences, Rijksuniversiteit Gent, Proeftuinstraat 86, 9000 Gent (Belgium)

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Neutron activation analysis (n.a.a.) coupled with Ge(Li) γ -ray spectrometry is a powerful method for the simultaneous determination of a number of trace elements in biological materials. Since high matrix activities prohibit the direct measurement of many isotopes, however, extensive separation schemes have been developed by several authors [1–8]; a chemical group separation combined with high-resolution γ -ray spectrometry [9, 10] offers the same possibilities. The chemical group separation outlined in this paper allows the determination of Ag, As, Cd, Co, Cr, Cs, Cu, Fe, Hg, K, La, Mn, Mo, Na, Rb, Sb, Se, Sc, Sn, and Zn; seventeen of these twenty elements can be determined within 5 d after irradiation. Accurate, rapid standardization is provided by the multi-isotopic ruthenium comparator.

STANDARDIZATION

The relative multiple comparator method, which has been described [11–14] elsewhere, eliminates the tedious chemical separations on a multi-element standard necessary to obtain an identical counting geometry, or avoids multiple measurements of single element standards (*cf.* ref.2). Determination of the infinite dilution resonance integrals [15, 16] extends the application of the method to a greater number of elements. Table 1 shows the ruthenium isotopes used as a comparator for the trace elements concerned. The specific activity of ^{105}Ru was calculated from the daughter activity ^{105}Rh , a case of non-equilibrium, as follows:

$$A_{\text{sp}}^{105}\text{Ru} = \frac{A^{105}\text{Rh}}{(1 - e^{-\lambda_{\text{Rh}}t}) \cdot e^{-\lambda_{\text{Rh}}t'} \cdot w} \cdot \frac{I_{\text{Ru}}}{I_{\text{Rh}}} \cdot \frac{\lambda_{\text{Ru}} - \lambda_{\text{Rh}}}{\lambda_{\text{Ru}}} \quad (1)$$

where $A_{\text{sp}}^{105}\text{Ru}$ = specific activity of ^{105}Ru expressed as counts $\text{mg}^{-1} \text{h}^{-1}$ saturation factor $^{-1}$ at the end of the irradiation;

$A^{105}\text{Rh}$ = measured activity of ^{105}Rh ;

*Research fellow of the I.W.O.N.L.

**Research associate of the I.I.K.W.

TABLE I

Nuclear data for the ruthenium isotopes and their respective use as comparator

Comparator		Classification of isotopes			
Target nuclide	Isotope formed by (<i>n</i> , <i>γ</i>)	Half-life	Natural abundance <i>θ</i> (%)	<i>γ</i> -energy of most important transition and absolute abundance (%)	<i>I</i> '/ <i>σ</i> '(<i>σ</i> ') Useful <i>I</i> ₀ / <i>σ</i> range
⁹⁶ Ru	⁹⁷ Ru	69.1 h	5.5	216 keV 11.7 %	23.1(0.25) 17 < <i>I</i> ₀ / <i>σ</i>
¹⁰² Ru	¹⁰³ Ru	39.5 d	31.6	496 keV 87.6 %	3.3(1.3) 0 < <i>I</i> ₀ / <i>σ</i> < 8.2
¹⁰⁴ Ru	¹⁰⁵ Ru ↓ <i>β</i> ⁻ ¹⁰⁵ Rh	4.44 h 35.5 h	18.6	316.5 keV 11.6 % 318.9 keV 19.3 %	13.0(0.47) 8.2 < <i>I</i> ₀ / <i>σ</i> < 17
		117mSn, 113Sn, 122Sb, 86Rb 38Cl, 51Cr, 60Co, 64Cu, 59Fe, 203Hg, 42K, 140La, 56Mn, 24Na, 46Sc, 69mZn, 65Zn 110mAg, 76As, 115Cd, 83Br, 134Cs, 99Mo, 75Se			

$\lambda_{\text{Ru}}, \lambda_{\text{Rh}}$ = decay constants of ^{105}Ru and ^{105}Rh ;

t = irradiation time;

t' = decay time;

w = weight of the comparator

I = fraction of emitted γ -rays per disintegration.

Care should be taken to avoid interference of the 316.5-keV photopeak of ^{105}Ru with the 318.9-keV peak of ^{105}Rh by allowing a sufficient decay time (e.g. 57 h waiting time for 10 h irradiation). One single measurement of ^{97}Ru , ^{103}Ru and ^{105}Rh about 3 d after irradiation thereby ensures the standardization of the 20 elements to be determined.

The k -factors were determined by irradiating 50- μl solutions of the elements (Table 2) spotted on 1.5 g of dextrose (analytical grade) packed in a polythene box (16 mm diameter, 21 mm high) and surrounded by six Ru comparators (each 10 mg). The irradiation time varied between 0.5 and 7 h. The organic dextrose dilution avoids self-shielding effects as with the biological sample. The low water content (max. 10 %) comparable with a lyophilized sample minimizes the possibility of a flux increase, through elastic neutron scattering with water protons [17]. The average flux ratio in the irradiation facility used is $\phi_{\text{th}}/\phi_{\text{epi}} = 32$. To determine the epithermal and thermal flux gradients, tin and copper discs (20 mm diameter; 0.15 mm thick) are irradiated and divided into 32 parts. The tin pieces are dissolved in 2 ml of 6 M hydrochloric acid and the 159-keV peak of $^{117\text{m}}\text{Sn}$ is measured on a Ge(Li) detector. The 511-keV β^+ -annihilation peak of the copper pieces is measured with a NaI(Tl) well-type crystal. The $^{117\text{m}}\text{Sn}$ activity is induced to an extent of 39 % by epithermal neutrons ($I_0/\sigma = 81$) and the ^{64}Cu activity to the extent of 4 % only ($I_0/\sigma = 1.3$). The average percentages of thermal and epithermal flux gradients, parallel to the reactor core, are 4 % cm^{-1} and 3 % cm^{-1} respectively. The radial average decrease of thermal and epithermal flux away from the reactor core is 4 % cm^{-1} and 13 % cm^{-1} respectively. Three ruthenium comparators were therefore placed above and beneath each sample to ensure a consistent mean value of flux and flux-ratios. Table 2 shows the k -values found experimentally for Ag, As, Cd, Co, Cr, Cs, Cu, Fe, Hg, La, Mn, Mo, Rb, Sb, Se, Sc, Sn and Zn. The k -factors of K and Na were determined four times with the Bowen Kale powder as a standard reference material. These values are also given, together with their reproducibility, in Table 2.

Simonits et al. suggested [18] the compilation of generalized k_0 factors, which are independent of irradiation and measuring conditions.

The k_0 -factor is defined by eqn.(2):

$$k_0 = \frac{M'}{M} \cdot \frac{\theta}{\theta'} \cdot \frac{\gamma}{\gamma'} \cdot \frac{\sigma}{\sigma'} = \frac{A_{\text{sp}}}{A'_{\text{sp}}} \cdot \frac{\epsilon'_p}{\epsilon_p} \cdot \frac{\phi_{\text{th}}/\phi_{\text{epi}} + I'_0/\sigma'}{\phi_{\text{th}}/\phi_{\text{epi}} + I_0/\sigma} \quad (2)$$

The parameters M' , θ' etc. refer to the comparator, and parameters M , θ etc. to the isotope.

ϵ_p = photopeak efficiency for the measured γ -ray energy;

M = atomic weight;

θ = isotopic abundance;
 γ = absolute abundance of the measured γ -ray;
 $A_{sp}/A'_{sp} = k$ = the experimentally determined k -value.

TABLE 2

Standardization

Element	Compound ^a	Solvent	Concn. $\mu\text{g}/50 \mu\text{l}$	Isotope	γ -Energy
Ag	AgNO ₃	H ₂ O	313.6	^{110m} Ag	657.5 884.5
As	As ₂ O ₃	0.4 M NH ₄ OH	13.65	⁷⁶ As	559 657
Cd	Metal	4.4 M HCl	23.09	¹¹⁵ Cd ^{115m} In	527 335
Co	Co ₂ O ₃	8.6 M HCl	463.7	⁶⁰ Co	1173 1332
Cr	Metal	1.2 M HCl	173.8	⁵¹ Cr	320
Cs	CsCl dried	H ₂ O	28.64	¹³⁴ Cs	569 796
Cu	Metal	0.8 M HNO ₃	45.35	⁶⁴ Cu	511
Fe	Metal	4.3 M HCl	970.8	⁵⁹ Fe	1098 1192
Hg	Metal	0.5 M HNO ₃			
La	La ₂ O ₃	0.7 M HNO ₃	361.9	¹³⁹ Hg ¹⁴⁰ La	279 328.7
		5.6 M HNO ₃	156.9		487 816 1596
Mn	MnO ₂	6 M HCl	104.11	⁵⁶ Mn	846
Mo	MoO ₃	1.4 M NH ₄ OH	17.63	⁹⁹ Mo— ^{99m} Tc	140
Rb	RbCl dried	H ₂ O	651.26	⁸⁶ Rb	1076
Sb	Metal (etched)	6 M HCl; 0.3 M HNO ₃	13.73	¹²² Sb	564
Se	Metal	0.7 M HNO ₃	535.6	⁷⁵ Se	120 136 265 279 400
		1.2% H ₂ O ₂			
Sc	Sc ₂ O ₃	12 M HNO ₃	1.274	⁴⁶ Sc	889 1120
Sn	Metal	12 M HCl	755.6	^{117m} Sn ¹¹³ Sn	159 391
Zn	Metal	1 M HCl	294.8	⁶⁵ Zn	1115
Na	Bowen's kale		2506 ^b	²⁴ Na	1368 2754
K	Bowen's kale		24615 ^b	⁴² K	1527.7

^a All materials were of p.a. grade.

^b In $\mu\text{g g}^{-1}$.

TABLE 2 (continued)

I_0/σ	Comparator	k -Factor	Found k_0	Theor. k_0
12.2	^{105}Ru	2345	174.8	165.9
		1383	139.7	132.4
9.5	^{105}Ru	2202	245.0	255.3
		232.4		
11.4-77	^{105}Ru	63.0	3.59	1.43-2.20
		173.2	5.87	2.38-3.71
2.0	^{103}Ru	2446	181.1	171.4
		2184	180.1	
0.5	^{103}Ru	45.9	Not determ.	
12.0	^{105}Ru	11309	381	307
		51547	2052	1930
1.3	^{103}Ru	132.2	3.99	4.96
1.4	^{103}Ru	0.1653	0.0113	0.0097
		0.1096	0.0086	0.0076
0.83	^{103}Ru	55.9	1.544	1.657
1.3	^{103}Ru	125.4	3.14	3.79
		193.9	7.00	8.31
		69.27	3.81	4.34
		144.8	13.8	17.52
1.0	^{103}Ru	1078	59.8	65.4
9.2	^{105}Ru	511.0	Not determ.	
7.9-17	^{97}Ru	44.67	3.389	2.49-5.67
33.6	^{97}Ru	4524	203	161
8.2	^{105}Ru	871.3	10.98	11.50
		2912	36.63	39.45
		1723	38.94	41.15
		757	16.75	17.31
		473	7.46	8.23
0.5	^{103}Ru	5830	167.1	157.9
		4688	168.9	
81	^{97}Ru	8.42	Not determ.	
38.6	^{97}Ru	10.31		
2.2	^{103}Ru	10.69	0.693	0.806
0.66	^{103}Ru	2.580 ± 0.029	1.191 ± 0.021	
		(1.12%)	(1.77%)	
0.90	^{103}Ru	0.04938 ± 0.00045		
		(0.91%)		

The first part of eqn.(2) can be calculated from known nuclear constants (M , θ , γ and σ), yielding a theoretical k_0 value. By measuring a relative efficiency curve for the Ge(Li) detector used and converting the detection efficiency of the sample and comparator geometries to a point source geometry at 12-cm distance from the detector, it is possible to calculate ϵ'_p/ϵ_p and thus the experimental k_0 -value. Both k_0 -values are given in Table 2. The relative efficiency curve was experimentally determined by activity measurement of the following point sources at a fixed distance of 12 cm from the detector, to

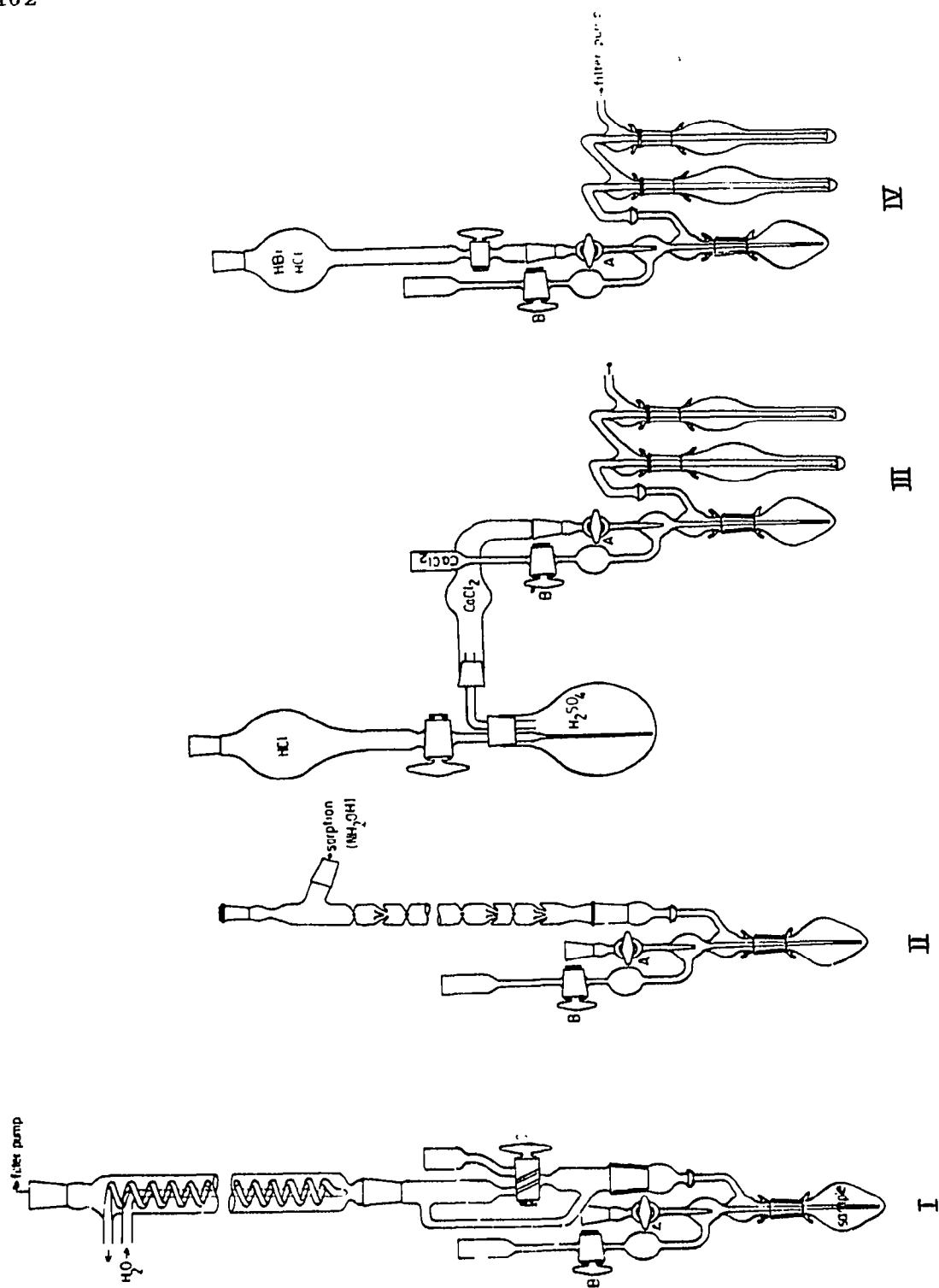


Fig. 1. Apparatus. I. Destruction. II. Bromine volatilization. III. CrO_3Cl_3 distillation. IV. As, Hg, Mo, Sb, Se, Sn distillation.

avoid 'real' coincidence of cascade γ -rays: ^{152}Eu , ^{154}Eu , ^{182}Ta , ^{133}Ba , ^{82}Br , $^{110\text{m}}\text{Ag}$, ^{75}Se . Additional measurements of some IAEA standard sources, e.g. ^{241}Am , ^{57}Co , ^{22}Na , ^{137}Cs , ^{54}Mn , ^{60}Co , allow the normalization of the relative curve to give an absolute curve for general use.

Once sufficient data are published, the general k_0 -values enable the calculation of k -values for a given comparator, a given reactor spectrum, and a given detector, as can be seen from eqn.(3):

$$k = k_0 \frac{\phi_{\text{th}}/\phi_{\text{epi}} + I_0/\sigma}{\phi_{\text{th}}/\phi_{\text{epi}} + I'_0/\sigma'} \cdot \frac{\epsilon_{\text{p}}}{\epsilon'_{\text{p}}} \quad (3)$$

All peak areas were determined by a suitable program [19] on a PDP 9 computer. In addition, a second program calculated specific activities of comparators, k_{anal} -values, an error estimate [13], a possible yield determination, and the element concentrations in routine analysis.

ANALYSIS OF SAMPLES

About 1.5 g of biological material was irradiated for 7 h in the Thetis reactor of the University of Ghent at a thermal neutron flux of $1.5 \cdot 10^{12} \text{ n cm}^{-2} \text{ s}^{-1}$. One hour after the end of the irradiation, a 30-mg sample is measured for ^{24}Na and ^{42}K activities (12 cm distance) with the Ge(Li) detector (useful volume 70 cm^3 ; energy resolution (FWHM) 2.0 keV for the 1332-keV peak of ^{60}Co).

The remaining sample is digested with 15 ml of 12 M HClO_4 and 8 ml of 14 M HNO_3 in a 50-ml distillation flask (Fig.1). Carrier (10 μg) is added for each of the elements, Ag, As, Cd, Co, Cs, Cu, La, Mn, Rb and about 100 μg for Sb, Se, Hg and Sn, all dissolved in nitric acid, with 10 mg of chromic acid in aqueous solution also present. At the same time known amounts of ^{77}As (prepared [20] on silica gel from irradiated germanium) and of ^{124}Sb tracer are added for yield determinations. The mixture is agitated by an air stream, created by a filter pump, at the top of the Nimroth water-cooler. The reaction receiver is heated by an electrically heated bath containing Woods metal; the temperatures recorded below were measured in this surrounding bath.

Primary oxidation of the biological material during digestion is achieved with nitric acid at 120°C for 15 min, whereupon the temperature is raised to 180°C and kept constant for another 5 min. Then the receiver is heated at 230°C with valve C closed. After about 5 min the green colour of the solution changes to orange-red ($\text{Cr(III)} \rightarrow \text{Cr(VI)}$), indicating complete destruction. Loss of tracers through volatilization does not occur [21]. The next stage (Fig.2) is the volatilization of bromine, performed by adding, dropwise, 5 ml of 48 % hydrobromic acid at 110°C during 15 min. Other possible volatile compounds reflux in the Vigreux column (Fig.1). The residual nitric acid and water is then distilled at 150°C and collected in the two-trap receiver. Possible losses during the volatilization of bromine and the removal of nitric acid and water amount

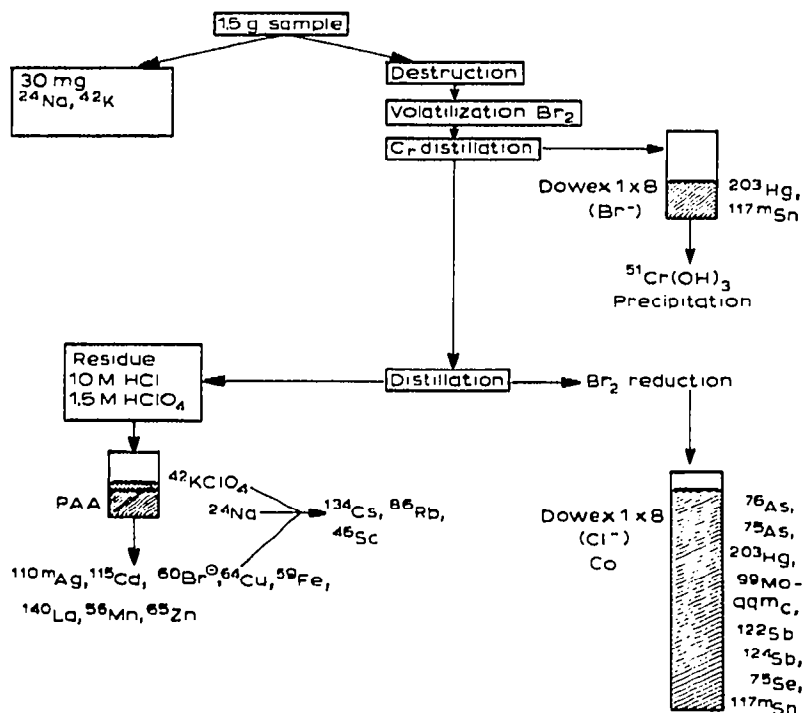


Fig. 2. Separation scheme.

to 3 % for Hg and to 0.7 % for As, Sb, Cr, Se, Sn and Mo. By raising the temperature to 210 °C and slowly introducing dried hydrogen chloride, CrO_2Cl_2 is distilled and collected in 30 ml of 48 % HBr —2 % NH_2OH (previously boiled for 1 min and cooled to room temperature). The two-trap receiver is ice-cooled. The distillation is repeated twice with 10 mg of CrO_3 carrier. Under these working conditions, more than 99 % of the As, Sb, Se and Mo remain in the distillation flask. However, the distillate appears to contain up to 40 % Hg and 55 % Sn which are now fixed on a 2-ml Dowex 1-X8 (200—400 mesh) column in the bromide form. Chromium(III) is not adsorbed and is precipitated as chromium hydroxide by adding 14 M ammonia solution up to pH 7, thus increasing the detection efficiency of the low-activity 320-keV γ -ray by a factor of 2 or 3. The quantitative distillation of As, Mo, Sb, Se and of the residual Sn and Hg is carried out by adding 100 ml of a mixture of HBr (48 %) and 12 M HCl (2 : 1) dropwise at 230 °C. Halfway through this distillation, 10 ml of 12 M HClO_4 is added to the distillation flask. Subsequently the bromine formed during the distillation is reduced to bromide by adding 8 g of hydroxyammonium chloride to the ice-cooled distillate and then heating gently in an Erlenmeyer flask fitted with a 60 cm-high spiral condenser. The six elements are next fixed on a 10-ml Dowex 1-X8 (200—400 mesh) column in the bromide, chloride form. The two resin fractions are combined, homogenized

a 20-ml counting vial, and dried by suction with a filter stick. The resin can then be measured for ^{77}As , ^{76}As , ^{203}Hg , ^{99}Mo — $^{99\text{m}}\text{Tc}$, ^{122}Sb , ^{124}Sb , ^{75}Se , $^{117\text{m}}\text{Sn}$ activities.

The distillation residue is evaporated to about 2 ml of HClO_4 and taken up again in hot 12 M HCl (15 ml). This solution is treated on a 1-g PAA (polyantimonic acid) column; sodium is completely retained and K, Cs, Rb and Sc are retained partially, while KClO_4 partially precipitates at room temperature. The following elements are eluted quantitatively from the PAA with 5 ml of 12 M HCl : Cu, Mn, Zn, Co, Ag, Fe, La, Cd. The eluate is measured for the first time (12 cm distance) with the Ge(Li) detector for ^{64}Cu and ^{56}Mn . The highly energetic γ -rays of the residual ^{42}K cause a dead-time of 15 %. This high counting-rate results in pile-up losses of up to 8 % as shown by tracer experiments. On the basis of Anders' hypothesis [22] that these losses are equal for all the γ -energies over the entire spectrum, a second measurement of ^{42}K a few days later (when the dead-time is less than 2 %) yields the net activity of ^{42}K , and comparison with the first measurement allows the calculation of the pile-up losses for the 846-keV ^{56}Mn peak and the 511-keV annihilation peak of ^{64}Cu (corrected for β^+ annihilation of ^{42}K).

After total decay of ^{42}K (about 5 d after irradiation), ^{115}Cd — $^{115\text{m}}\text{In}$, ^{140}La , ^{59}Fe , ^{60}Co , ^{65}Zn and $^{110\text{m}}\text{Ag}$ are measured in the PAA eluate. After total decay of ^{24}Na (ca. 14 d), the PAA column is added to the PAA eluate and the solution is evaporated to dryness so that the quantitative measurement of ^{86}Rb , ^{46}Sc and ^{134}Cs becomes possible.

TABLE 3

Yield determinations tested by tracer experiments
(Two determinations were done unless otherwise specified)

Element	Yield (%)	Remarks
Rb ^a	99	80—92 % on PAA
Cd, Cu, Mn ^a , Se ^b , Sc, Zn	98	Av. 88 % on PAA for Sc
Co ^a , Cs, Cr, La, Sn	96	91 % on PAA, 5 % in eluate for Cs
Ag, Fe, Mo	94	
As ^c	93	
Hg	90	
Sb ^c	80	80—95 % irreproducible

^a 3 determinations.

^b 5 determinations.

^c 4 determinations.

RESULTS AND DISCUSSION

The yields for the 18 elements were tested by tracer experiments in the presence of 1.5 g of lyophilized pork liver (Table 3). The values are satis-

TABLE 4

Bowen's kale powder

Element	Found (p.p.m.)		Mean value (p.p.m.)		s_T (total) (%)	s_T (count) (%)	Best mean values or ranges [23]	
Ag	< 0.04						< 0.01-0.5	
As	0.134	0.127	0.122		6.0	2.4	0.14 (0.11-0.22)	
Cd	1.07	0.95	0.90	1.07	8.3	3.5	0.80 (0.38-1.06)	
Co	0.058	0.065	0.063	0.057	8.6	7.3	0.058 (0.04-0.08)	
Cr	0.452	0.430	0.462	0.448	2.8	1.4	0.31 (0.18-0.42)	
Cs	0.084	0.081	0.078	0.072	6.7	4.7	0.074 (0.069-0.77)	
Cu	5.4	5.7	5.2	5.3	4.6	1.8	5.0 (3.6-6.5)	
Fe	104	104	106	102	3.1	2.4	118 (88-157)	
Hg	0.142	0.147	0.149		3.4	3.0	0.167 (0.11-0.23)	
La	0.1073	0.1061	0.1062	0.1061	0.91	1.6	0.087 (0.08-0.1)	
Mn	15.2	16.3	15.9	16.7	4.8	0.30	14.7 (12.6-18)	
Mo	1.93	2.07	1.97	2.04	4.3	0.54	2.28 (1.5-3.1)	
Rb	48.6	48.0	51.4	47.3	3.4	0.49	52.2 (41-57)	
Sb	0.065	0.058	0.059	0.068	9.5	3.2	0.069 (0.05-0.11)	
Se	0.123	0.116	0.118	0.106	6.9	6.7	0.121 (0.02-0.15)	
Sc	0.0120	0.0103	0.0104	0.0111	6.9	1.6	0.008	
Sn	< 0.6						0.26	
Zn	31.1	31.2	33.7	32.4	3.7	1.0	33.2 (30-38)	

factory with the exception of those for antimony, which required an independent yield determination. The good reproducibility and accuracy of the method can be seen from the 6 analyses of Bowen's kale powder [23] (Table 4) and the 2 analyses of NBS Bovine Liver, SRM-1577 (Table 5). The dry weight appeared to be 94.3 % for Bowen's kale powder (20 h drying at 90 °C) and 94.6 % for the Bovine Liver SRM-1577 (24 h freeze-drying). The chromium result for Bowen's kale is rather high, although the precision is excellent. The difference between the two chromium results for SRM-1577 may indicate inhomogeneity for this element. Interferences from $^{54}\text{Fe}(n,\alpha)^{51}\text{Cr}$, $^{56}\text{Fe}(n,p)^{56}\text{Mn}$ are negligible. According to Chauvenet's criterion [24] the fifth result for the lanthanum concentration in Bowen's kale powder was omitted from the mean value.

TABLE 5

Determination of trace elements in bovine liver
($\mu\text{g g}^{-1}$ dry weight)

Element	(67 % conf. lim.)	NBS value ^a	Element	(67 % conf. lim.)	NBS value ^a
Ag	0.051 ± 0.011	—	Mn	11.2 ± 0.5	10.3 ± 1.0
As	0.056 ± 0.004	(0.055)	Mo	3.19 ± 0.14	(3.2)
Cd	0.288 ± 0.035	0.27 ± 0.04	Rb	17.97 ± 0.60	18.3 ± 1.0
Co	0.223 ± 0.011	(0.18)	Sb	0.0048 ± 0.0005	—
Cr	0.074 ± 0.005	—	Se	1.02 ± 0.03	1.1 ± 0.1
	0.123 ± 0.006		Sc	< 0.0005	—
Cu	187 ± 8	193 ± 10	Sn	< 0.6	—
Cs	0.0149 ± 0.0022	—	Zn	135 ± 5	130 ± 10
Fe	236 ± 5	270 ± 20	Na	2632 ± 29	2430 ± 130
Hg	0.0223 ± 0.0013	—	K	10323 ± 258	9700 ± 600
La	0.0173 ± 0.0004	—			

^a Values in parenthesis are not certified.

The standard deviation for a single measurement, calculated from the six results, determines the overall precision for one analysis. The error from the analytical treatment (assuming that the material is homogeneous) was calculated from the average counting error for a single analysis and from the overall precision.

In conclusion, the present method of trace analysis is applicable to any biological material, and is versatile, simple, and reproducible. A skilled analyst requires about five hours to perform all the required chemical manipulations after irradiation. The measurements of some γ -activities are rather time-consuming. Nevertheless, quantitative results for 20 elements are available within two weeks with the aid of ruthenium as a multi-comparator; this includes the processing of the data with a suitable computer program.

SUMMARY

A separation scheme for the determination of 20 trace elements by neutron activation analysis has been developed and tested on biological material. The method is based on a wet chemical destruction followed by distillation, ion exchange separation and column chromatography (polyantimonic acid) for the removal of the sodium matrix activity. The recovery of tracers added to liver samples has been studied for the following elements: Ag, As, Cd, Co, Cs, Cu, Cr, Fe, Hg, La, Mn, Mo, Rb, Sb, Se, Sc, Sn and Zn. The yields exceeded 90 % except for Sb. The accuracy and reproducibility can be deduced from repeated analyses of Bowen's kale powder and from the NBS bovine liver, standardised by the relative multiple comparator method. The reproducibility varied between 0.91 % and 9.5 % (counting statistics included).

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