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**REFERENCE MATERIAL FOR TRACE  
ANALYSIS BY RADIOANALYTICAL  
METHODS: BOWEN'S KALE**

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REFERENCE MATERIAL FOR TRACE ANALYSIS BY RADIOANALYTICAL  
METHODS: BOWEN'S KALE

IUPAC

Analytical Chemistry Division

Commission V. 7 for Analytical Radiochemistry and Nuclear Materials

Abstract - A fairly large volume of published data on 'Bowen's Kale' has been examined critically in order to develop recommendations for the use of this preparation as a 'reference material' in the standardisation and evaluation of the reliability of analytical procedures. Values are now recommended for the contents of twelve elements present in major to trace concentrations in 'Bowen's Kale'. 'Indicated values' for another 16 elements are provided. Values for 15 more elements are listed with no recommendation. The criteria adopted in categorising elements into these groups are discussed.

#### INTRODUCTION

The increasing interest in the examination of biological materials for trace elements (Refs. 1-8) has focussed attention on the development of reference materials for the standardisation and evaluation of the reliability of analytical procedures and data (Refs. 9-14). It is recognised that standards containing different concentration of major, minor and trace elements are needed. However, the number of such standards currently available from internationally accepted Standards Organisations is relatively small (Refs. 15-18).

The IUPAC Commission for Analytical Radiochemistry and Nuclear Materials has now examined the data available on 'Bowen's Kale', referred to hereafter as 'Kale', with a view to consider its suitability as a reference material for radiochemical analyses of biological samples. The material consists of dried leaves of Kale (*Brassica Oleracea*) converted to a powder, without the use of metal tools. It was prepared and distributed by Prof. H. J. M. Bowen, The University of Reading, U. K., in 1964 (Ref. 11). Since then, it has been analysed in a large number of laboratories by different methods. The original stock of 'Kale' was about 90 kg, of which a significant fraction is still available from the original source.

On the basis of an examination of the published data on this material, the Commission recommends the use of 'Kale' as a reference material for radioanalytical investigations of biological samples.

#### HOMOGENEITY

The published data on 'Kale' (Refs. 9 & 11) and the results obtained by members of the Commission (Refs. 19 & 20) show that 'Kale' samples of more than 100 mg can be considered homogeneous for the elements listed in Tables I and II of this report. It is therefore advised that a sample of at least this size should be used in each determination. The water content of 'Kale' is about 5% and it is recommended that the dry weight of 'Kale' be determined by drying at 90°C for 20 h. (Ref. 17).

#### ELEMENTS CHARACTERISED

The elements characterised in 'Kale' are categorised into three groups.

## ELEMENTS WITH RECOMMENDED VALUES

Table I lists the concentrations of elements for which 'Kale' is recommended as a reference material. The following method was used to compute the value for an element:

If there were more than ten 'laboratory means' for an element and if more than one technique was employed for the analysis, a grand mean and the standard deviation of this mean were computed. Obvious 'rogue values' were excluded in computing the mean. Those 'laboratory means' which deviated from the grand mean, as calculated above, by twice the standard deviation were eliminated in calculating the 'recommended mean and the standard deviation values' listed in Table I.

Additional criteria for including elements in Table I were that (a) there were at least 10 acceptable laboratory means, as defined above, for calculating the 'recommended value' and that (b) the coefficients of variation were less than 5, 10 and 20 per cent for elemental concentrations above 100, above 1 and below 1 ppm, respectively.

## ELEMENTS WITH INDICATED VALUES

Table II lists elements which fall into one of the following groups:

- (a) Elements that were determined by more than the minimum number of laboratories, but did not have at least 10 acceptable values, or were not determined with the precision required for "elements with recommended values" (see above).
- (b) Elements for which there were less than the required number of acceptable means but for which all values fell in a reasonably close range. In all such cases the 'indicated value of the mean and standard deviation' are given in Table II. It is to be understood that the values given are good estimates of the concentrations of those elements in 'Kale'.

## ELEMENTS FOR WHICH THERE IS NO RECOMMENDATION

For the elements listed in Table III the limited number and the spread of available data point to the need for exercising caution in accepting a value for reference purposes.

The Commission wishes to encourage further work on 'Kale' to improve its characterisation, considering that it is available in relatively large amounts and that the work done already covers a major portion of the total effort needed to develop a reference material for radioanalytical investigations of biological samples.

TABLE I. Values for elements for which 'Kale' is recommended as a reference material.

Element	Range of values used for calculations*	Recommended value**
A. Major constituents (% by weight)		
Calcium	3.80 - 4.50(22;6)	4.14 ± 0.18
Potassium	2.22 - 2.70(17;1)	2.43 ± 0.13
B. Minor constituents (% by weight)		
Magnesium	0.142 - 0.170(16;4)	0.156 ± 0.008
C. Trace constituents (ug/g)		
Iron	103 - 123.2(18;4)	115 ± 6
Rubidium	41 - 59.6(10;0)	52 ± 5.4
Zinc	26.7 - 34(26;4)	31 ± 2.2
Bromine	23.1 - 26(12;2)	24 ± 1.4
Manganese	12.2 - 17(38;6)	15 ± 1.2
Copper	4.12 - 5.63(20;4)	4.9 ± 0.42
Molybdenum	1.9 - 2.59(18;2)	2.3 ± 0.21
Mercury	0.15 - 0.225(15;5)	0.18 ± 0.03
Cobalt	0.041 - 0.083(19;3)	0.06 ± 0.01

\* The first number in brackets represents the number of the laboratory means examined whereas the second shows the number of results rejected. Range does not include the rejects.

\*\* Value on dry sample; for procedure see Ref. 11 or 17.

The ± values are the 'root mean square of the deviations of the laboratory means'.

TABLE II. Elements with 'indicated values' in 'Kale'.

Element	Range of values used for calculations*	Indicated value**
A. Major constituent (% by weight)		
Nitrogen	4.10 - 4.60(6;0)	4.33 ± 0.16
B. Minor constituents (% by weight)		
Phosphorus	0.402 - 0.470(5;0)	0.445 ± 0.026
Chlorine	0.30 - 0.398(11;1)	0.35 ± 0.03
Sodium	0.201 - 0.284(16;3)	0.230 ± 0.023
C. Trace constituents (ug/g)		
Boron	41.2 - 50.9(5;1)	46 ± 4
Cadmium	0.79 - 1.056(12;4)	0.89 ± 0.09
Vanadium	0.32 - 0.414(7;1)	0.36 ± 0.04
Tin	0.16 - 0.25(9;3)	0.21 ± 0.03
Arsenic	0.114 - 0.18(12;2)	0.14 ± 0.02
Selenium	0.131 - 0.16(12;3)	0.14 ± 0.01
Antimony	0.0615 - 0.0945(9;2)	0.07 ± 0.01
Lanthanum	0.06 - 0.1(6;1)	0.08 ± 0.01
Caesium	0.0688 - 0.0807(5;0)	0.075 ± 0.005
Tungsten	0.059 - 0.0607(4;0)	0.06 ± 0.007
Scandium	0.006 - 0.0084(8;2)	0.008 ± 0.0009
Gold	0.00206 - 0.00294(8;0)	0.0023 ± 0.0004

\* See footnote under Table I.

\*\* These values are good estimates.

TABLE III. Elements with no recommended values in 'Kale'.

Element	Values reported(ug/g)	Remarks
Sulphur	13700, 16010, 19800, 24770	) Poorly characterised at this level of concentration
Silicon	130, 233, 242	
Strontium	61, 65, 74.7, 76, 83, 88.1, 100.2, 101, 103, 122, 149.6	
Aluminium	6.4, 7.35, 35.5, 41, 41, 43, 45, 49, 60.9, 78.5, 80.1	Three groups of values; large scatter of results obtained by the same technique and the same worker.
Fluorine	4.92, 5.55, 6.45, 6.69	) Too few results
Barium	4.38, 6.4, 7.9	
Lead	1.6, 2.1, 2.2, 2.425, 2.65, 2.7, 3, 3.43, 3.8, 5.4	) Poorly characterised
Chromium	0.25, 0.285, 0.331, 0.332, 0.38, 0.43, 0.5, 0.67, 1.54	
Nickel	0.28, 1.08, 1.1, 2.65, 10.98	
Silver	0.0295, 0.5, 0.5, 0.5	
Titanium	0.33, 2.75	
Cerium	0.14, 0.457	
Iodine	0.08	
Gallium	0.045	
Europium	0.012, 0.004, 0.0066	

APPENDIX - Sources of the data used in developing  
the above recommendations.

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