

TRACE ELEMENTS IN THE COMMON BROWN ALGAE AND IN SEA WATER

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INTRODUCTION

Sea water probably contains all the chemical elements, although a number of them have not yet been detected. Considerable information is available on the occurrence of various elements in marine plants and animals, but it is far from complete for any one biological group. Except for recent work by Spooner (1949), on the absorption of radioactive strontium and yttrium by marine algae, no detailed study of the trace elements in the brown algae, nor any attempt to correlate the trace elements in sea water with those in the algae, appears to have been carried out. The following is a brief résumé of the work that has been done in this field.

The distribution and concentration of iodine in the sea have received a great deal of attention because of their important roles in the physiology of man and terrestrial animals. The form in which iodine occurs in sea water is not clearly understood, but part of it is present as iodide and iodate. It is concentrated to a marked degree by the marine algae, and for many years the Phaeophyceae were used as a commercial source of iodine. The concentration of iodine and its seasonal variation in the brown marine algae have already been reported (Black, 1948*a, b*, 1949).

Considerable interest has recently been shown in the content of radioactive elements in sea water. Studies by Evans, Kip & Moberg (1938), and by Föyn, Karlik, Pettersson & Rona (1939), have shown that the radium content measured by the radon emanation technique varies between about 0.2 and 3.0×10^{-7} $\mu\text{g./l.}$ in sea water of salinity approximately 35‰. These workers found that organisms concentrate the radium about one-hundredfold in their soft tissues, and calcareous structures show an increase in the radium : calcium ratio over that in the water. In a search for the radioactive precursors of radium, Karlik analysed a number of samples from various parts of the oceans and obtained a mean value of 1.5 $\mu\text{g./l.}$ for uranium (Föyn *et al.* 1939).

Tables giving the trace elements which have so far been determined in sea water have been compiled by Sverdrup, Johnson & Fleming (1942, pp. 176-7), Harvey (1945, pp. 31-2), and Legendre (1947).

In 1919, Cornec carried out the first spectrographic analysis of marine plants and found the following elements: Ag, As, Co, Cu, Mn, Ni, Zn, Bi, Sn, Ca, Mo, Au, Sb, Ti, W and U. The analysis, however, was qualitative and the species investigated were not given.

The distribution of arsenic in marine algae has been studied by Jones (1922), and by Williams & Whetstone (1940). Jones determined the arsenic content of eleven varieties of British seaweeds and obtained a value of 125 mg./kg. for *Laminaria digitata*.

The titanium in a number of cryptogamic plants has been estimated by Bertrand & Voronca-Spirt (1930), while Meulen (1932) determined molybdenum in several species of algae and reported a figure of 0.16 mg. Mo/kg. dry weight.

Webb (1937), in his studies on the ultimate composition of biological material, carried out a spectrographic analysis of marine invertebrates and included the three algae, *Ulva lactuca* (frond), *Fucus serratus* (receptacles) and *Saccorhiza bulbosa* (stipes). Figures are given for Na, K, Ca, Mg, Sr, Ba, B, Al, Mn, Fe, Cu and Pb.

In 1938, Igelsrud, Thompson & Zwicker found the boron content of five marine algae to be 4.2-14.9 mg. B/kg. dry material, and Lagrange & Tchakirian (1939) examined *Lithothamnium* and detected spectrographically in the ash, apart from ordinary constituents, Ag, As, Be, Cu, Ge, Mn, Mo, Ni, Pb, Sb, Sn, Ti, U, W and Zn. The rare earths in similar algae have been examined by Servigne & Tchakirian (1939), who analysed the ashes of *L. calcareum* and found about 5 mg./kg. of Pr, Nd and Sm.

The radium content of a number of algae has been determined by Wiesner (1938). Seven fresh-water and sixteen salt-water algae from different localities were examined, and the results indicated considerable accumulation of radium by the plants.

In 1940, Öy discussed the physiological importance of iron, copper, manganese and boron, and determined these elements in several species of algae. Iron varied between 120 and 1330 mg./kg. dry matter, while *Ascophyllum nodosum* contained 1.1-1.4 mg./kg. copper, *Laminaria* 4 mg./kg., *Fucus serratus* 5.8-17.4 and *F. vesiculosus* 3.4-8.4 mg./kg. Manganese was present in the Fucaeae to the extent of 100-130 mg./kg., and boron 100 mg./kg.

A spectrographic analysis of a dried seaweed meal carried out by the Macaulay Institute for Soil Research was reported by Beharrell in 1942; and in 1941 Wilson & Fieldes estimated spectrographically eighteen of the minor elements of *Macrocystis pyrifera*. No further work appears to have been carried out since that time.

EXPERIMENTAL

With the exception of *Laminaria cloustoni*, which was collected at Cullipool, Luing Island, Argyllshire, and one sample of *L. digitata* from Ardencaple

Bay, Seil Island, Argyllshire, all the samples of algae were collected at Atlantic Bridge, which joins the Island of Seil with the mainland of Scotland.

Immediately after collection the samples were air-dried at 25–30° C. for 48 hr., with every precaution taken to prevent metallic contamination and, with the exception of the last two sets of samples, were milled in a small porcelain-edge runner mill. With the later samples, however, the milling was carried out in a Christy and Norris no. 8 laboratory mill.

The sea-water samples were collected 20 miles off Plymouth¹ at 20 m., and from $\frac{1}{2}$ m. at Atlantic Bridge and Ardencaple Bay. In the first series of experiments the samples of sea water were stored both in Pyrex and Polythene bottles, but the analyses were identical, thereby indicating that no difference in composition has resulted from diatoms adhering to the glass surface or from the straight surface adsorption of trace elements on the glass. Pyrex bottles were therefore used for the remainder of the samples.

In Table I, the washed frond of *L. digitata* was obtained by soaking the whole frond in 5 gallons of ordinary tap water for 24 hr., prior to air-drying and milling.

ANALYTICAL METHODS USED

The trace-element analyses reported in this paper were carried out at the Macaulay Institute for Soil Research by a cathode layer arc spectrochemical technique, differing only in detail from that employed for plant materials (Mitchell, 1948). For a few elements semi-quantitative assessments only were made, but unless specifically indicated the results were obtained by a quantitative technique: directly on the ashed material (Farmer, 1950) for Cu, Ba, Sr, Mn, and by a method involving chemical concentration prior to spectrographic analysis (Mitchell & Scott, 1948) for Co, Ni, Mo, Fe, Pb, Sn, Zn, V, Ti, Cr and Ag. Even after concentration of the 100-fold order one or two of the elements reported, notably Sn, Ag, and in a few instances Pb, are close to the lower limit of determination, and these results may be somewhat less reliable than those for the other elements.

DISCUSSION OF RESULTS

As with the major constituents of seaweed the results indicate that the content of trace elements depends on the species and on the stage of development, and that there is probably appreciable seasonal variation.

¹ The following determinations have been made on the 20 m. sample collected off Plymouth:

	Determination	Observer or analyst
Temperature	9.28° C.	Mr F. A. J. Armstrong
Salinity	35.12 ‰	Government chemist
Inorganic phosphate	16.0 µg./l. P	Mr F. A. J. Armstrong
Total phosphorus	19.2 µg./l. P	Mr F. A. J. Armstrong

No doubt some variation will occur in different habitats, where natural deposits and land drainage may influence the trace elements in the inshore waters where the algae are growing, but this has not been investigated.

It has already been shown (Black, 1948*a, b*, 1949) that iodine varies considerably in the different species and even in the same species, showing a progressive increase with the depth of immersion of the plant, increasing, for example, from 0.03% (dry basis) in *Pelvetia canaliculata* and *Fucus spiralis* to over 1% in *Laminaria cloustoni*. Although the seasonal variation is somewhat erratic, the iodine, which exists both in the organic and inorganic form,

TABLE I. TRACE-ELEMENT CONTENTS OF *LAMINARIA* FRONDS IN MG./KG. OVEN-DRIED MATERIAL

	<i>Laminaria digitata</i> frond. Collected July 1948, unwashed	<i>Laminaria digitata</i> frond. Collected July 1948, washed	<i>Laminaria cloustoni</i> frond. Collected Oct. 1947, unwashed
Total ash (% of dry matter) ...	24.2	11.8	13.8
Estimations quantitative			
Co	0.21	0.31	0.21
Ni	1.7	1.40	0.9
Mo	0.16	0.21	0.16
Pb	—	20	13
Sn	3.8	1.0	1.0
Zn	148	93	59
V	0.4	1.9	1.1
Ti	3	52	21
Cr	1.5	1.8	0.9
Ag	0.2	0.2	0.2
Fe	292	402	314
Estimations semi-quantitative			
Cu	120	70	30
Rb	25	80	30
Li	1	4	2
Ba	80	100	30
Sr	400	800	400
Mn	12	30	15

is generally at a maximum when the total ash and crude proteins are at a maximum. With the trace elements the reverse is true. In general, the trace elements are higher in May than in January and there appears to be no correlation between the trace-element content and the total mineral matter. If the phenomenon of trace-element concentration is one of ion exchange (Wassermann, 1949), it is reasonable to find that the concentration varies with the stage of development of the plant. The stipe which is perennial is consequently higher in these elements than the frond. It may also be, however, that the trace elements in the frond are utilized in sporogenesis, etc. It is interesting to find, therefore, that many of the trace elements such as Co, Ni, Mo, Fe, etc. (Table II), which may be essential for reproduction, are lower in a sporing than in a sterile frond.

Soaking a fresh *Laminaria* frond in water, although it removes a considerable amount of the water soluble mineral matter presumably in solution in the cell sap, does not remove the trace elements, which appear, therefore, to be in an insoluble form, possibly adsorbed on the colloids present, or combined with the polysaccharides such as alginic acid or fucoidin (Table I).

Samples of the various species collected in January 1949 (Table II) show considerable variation in trace-element contents, and *Fucus serratus* appears to be exceptional in concentrating iron to 717 mg./kg. and manganese to 800 mg./kg.

A comparison of the trace elements in a sporing and non-sporing frond of *Laminaria cloustoni* shows slight differences, and it may be, therefore, that the trace elements are utilized to some extent in reproduction.

A further set of samples taken in May 1949 shows that on the anhydrous basis the trace elements undergo quite appreciable seasonal variation. Despite the fact that the total ash has decreased the trace-element content shows considerable increases (Table III), e.g. in *Pelvetia canaliculata* nickel increases from 1.9, iron from 195, and titanium from 11.4 mg./kg. in January 1949, to 3.7, 565 and 37.8 mg./kg. respectively in May.

The samples taken in June 1950 (Table IV) differ quite appreciably from those taken in May 1949 (Table III). With the exception of *Fucus serratus*, the iron content, for example, is considerably higher in the 1950 samples, but this might be due to metal contamination, as the 1950 samples were milled in a C. and N. Mill, while the others were ground in a porcelain-edge runner mill.

The results in Table IV again show the differences which occur in the trace-element content of different species from the same habitat, with *F. spiralis* unaccountably high in most of the trace elements, for example, 308 mg./kg. of titanium as compared with 4 mg./kg. in *Laminaria digitata* frond. At this time of the year the dry-weight content of *Fucus spiralis* is very low, the plant having taken in large volumes of water preparatory to shedding its gametes. This is further evidence of a correlation which may exist between the trace-element content and the reproductive cycle of the plant. Even when the results are calculated on the wet basis (Table V) *F. spiralis* still contains higher concentrations of the trace elements, particularly titanium and iron, than the Laminariaceae.

Despite the fact that one of the samples of sea water was taken in February, 20 miles off Plymouth at a depth of 20 m., and the other from inshore waters at Ardencaple Bay and Atlantic Bridge in June, the differences in the trace elements estimated are quite insignificant, but the contents are higher than those recorded in the literature (Table VI).

In Table VII the 'concentration factor' shows the extent to which marine algae can concentrate the trace elements, *F. spiralis* containing 10,000 times more titanium than the surrounding sea water, although the majority of the factors are smaller.

TABLE II. TRACE-ELEMENT CONTENTS OF OVEN-DRIED SEAWEEDS (COLLECTED 12 JANUARY 1949) IN MG./KG.

Sample	Total ash (% of dry matter)	Trace elements (mg/kg)																
		Co	Ni	Mo	Fe	Pb	Sn	Zn	V	Ti	Cr	Ag	Rb	Li	Sr	Ba	Mn	Cu
<i>Pelvetia canaliculata</i>	24.6	0.37	1.9	0.34	195	4	0.8	40	1.2	11	0.6	0.2	100	6	> 2400	70	70	5
<i>Ascophyllum nodosum</i>	27.2	0.41	1.5	0.69	168	6	1.0	103	1.9	9	0.7	0.3	80	4	2600	50	50	4
<i>Fucus serratus</i>	27.4	0.47	1.6	0.40	717	21	1.2	79	3.3	9	2.6	0.6	170	6	> 2800	80	800	11
<i>Laminaria digitata</i> frond	41.5	1.46	8.2	0.32	350	13	0.7	99	2.0	20	1.1	0.4	240	8	4000	120	80	20
<i>L. digitata</i> stipe	39.7	0.43	3.2	0.15	N.d.	N.d.	N.d.	N.d.	2.2	16	1.4	0.2	240	4	4000	60	100	16
<i>L. cloustoni</i> frond, sterile	32.4	0.56	2.0	0.50	283	10	0.7	117	1.3	19	1.2	0.7	250	6	3000	60	30	—
<i>L. cloustoni</i> frond, sporing	22.4	0.35	1.5	0.24	226	26	1.0	136	1.0	20	1.2	0.5	130	4	2200	65	30	15

TABLE III. TRACE-ELEMENT CONTENTS OF OVEN-DRIED SEAWEEDS (COLLECTED 26 MAY 1949) IN MG./KG.

Sample	Total ash (% of dry matter)	Trace elements (mg/kg)															
		Co	Ni	Mo	Fe	Pb	Sn	Zn	V	Ti	Cr	Ag	Sr	Ba	Mn	Cu	
<i>Pelvetia canaliculata</i>	21.64	0.72	3.7	0.35	565	5	1.3	47	2.6	38	1.2	0.2	> 700	20	22	5	
<i>Fucus spiralis</i>	24.34	1.39	6.0	0.29	638	5	1.8	62	1.9	27	0.9	0.2	> 700	19	104	6	
<i>Ascophyllum nodosum</i>	19.49	0.73	4.4	0.29	283	4	0.7	60	1.5	26	1.0	0.2	> 700	13	27	4	
<i>F. serratus</i>	21.77	0.84	3.2	0.65	375	10	1.3	70	2.0	20	0.7	0.3	> 700	22	155	5	
<i>F. vesiculosus</i>	23.97	0.65	3.8	0.34	221	2	0.5	60	1.9	28	1.8	0.2	> 700	44	116	7	
<i>Laminaria digitata</i> frond	31.84	0.29	2.4	0.19	138	2	1.0	64	0.6	5	0.4	0.3	> 700	13	9	< 3	
<i>L. digitata</i> stipe	—	0.92	3.9	0.10	293	6	1.7	62	0.3	2	0.4	0.4	> 700	15	10	5	
<i>L. cloustoni</i> frond	32.16	0.25	1.6	0.14	159	12	1.4	76	0.9	10	1.5	0.3	> 700	31	10	14	

TABLE IV. TRACE-ELEMENT CONTENTS OF OVEN-DRIED SEAWEEDS (COLLECTED 27 JUNE 1950) IN MG./KG.

Sample	Total ash (% of dry matter)	Co	Ni	Mo	Fe	Pb	Sn	Zn	V	Ti	Cr	Ag	Sr	Ba	Mn	Cu
<i>Pelvetia canaliculata</i>	23.3	1.30	4.8	0.55	2040	13	2.2	90	3.2	60	1.2	0.3	720	34	51	16
<i>Fucus spiralis</i>	22.8	2.00	9.3	1.32	3380	5	N.d.	N.d.	11.9	308	3.7	0.4	420	64	121	31
<i>Ascophyllum nodosum</i>	20.4	0.73	3.7	0.89	1150	4	1.1	116	2.8	28	1.9	0.1	570	18	36	12
<i>F. vesiculosus</i>	19.5	0.91	5.9	0.34	730	7	1.1	105	1.7	27	1.5	0.2	730	22	102	10
<i>F. serratus</i>	18.0	0.63	4.5	0.21	320	4	0.5	63	0.6	7	0.7	0.2	520	16	120	6
<i>Laminaria digitata</i> frond	26.2	0.31	1.8	0.16	400	7	0.6	59	0.5	4	1.1	0.1	690	17	< 30	5
<i>L. digitata</i> stipe	38.9	0.46	3.7	0.28	1260	16	2.8	92	0.7	8	1.8	0.4	1150	28	< 30	11
<i>L. digitata</i> frond, Ardencaple Bay	29.7	0.22	2.1	0.17	410	4	0.2	71	0.7	4	1.8	0.0	950	18	< 30	6
<i>L. digitata</i> stipe, Ardencaple Bay	37.8	0.62	5.7	0.34	1570	7	0.2	85	1.2	5	2.9	0.0	1120	20	< 30	21

TABLE V. TRACE-ELEMENT CONTENTS OF WET SEAWEEDS (COLLECTED 27 JUNE 1950) IN MG./KG.

Sample	Co	Ni	Mo	Fe	Pb	Sn	Zn	V	Ti	Cr	Ag	Sr	Ba	Mn	Cu
<i>Pelvetia canaliculata</i>	—	1.1	0.14	473	3.0	0.51	21	0.74	14	0.28	0.07	167	7.9	12	3.7
<i>Fucus spiralis</i>	0.37	1.7	0.24	622	0.92	N.d.	N.d.	2.2	57	0.68	0.07	77	12	22	5.7
<i>Ascophyllum nodosum</i>	0.17	0.92	0.22	285	1.00	0.27	29	0.69	6.9	0.47	0.03	141	4.5	8.9	2.9
<i>F. vesiculosus</i>	0.21	1.36	0.07	169	1.6	0.25	24	0.39	9.2	0.35	0.05	169	5.1	24	2.3
<i>F. serratus</i>	0.12	0.87	0.04	62	0.78	0.10	12	0.12	1.4	0.14	0.04	101	3.1	23	1.2
<i>Laminaria digitata</i> frond, Atlantic Bridge	0.04	0.25	0.03	56	0.97	0.08	8.2	0.07	0.56	0.15	0.01	96	2.4	< 4	0.7
<i>L. digitata</i> stipe, Atlantic Bridge	0.06	0.47	0.04	160	2.0	0.36	11.7	0.09	1.0	0.23	0.05	146	3.6	< 3.8	1.4
<i>L. digitata</i> frond, Ardencaple Bay	0.03	0.36	0.03	71	0.69	0.03	12.2	0.12	0.69	0.31	0.0	163	3.1	< 5.2	0.9
<i>L. digitata</i> stipe, Ardencaple Bay	0.07	0.64	0.03	176	0.78	0.24	9.5	0.13	0.56	0.32	0.0	125	2.2	< 3.3	2.4

TABLE VI. TRACE-ELEMENT CONTENTS OF SEA WATER IN $\mu\text{G. PER LITRE}$

Sample	Co	Ni	Mo	Pb	Sn	Zn	V	Ti	Cr	Ag	Sr	Ba	Mn	Cu
Taken 20 miles off Plymouth at 20 m. on 22. ii. 50. Stored in Pyrex bottle	<0.3	6	12	<8	<5	9	2.4	8	1.2	2.9	N.d.	N.d.	N.d.	N.d.
As in (1), but stored in Polythene bottle	<0.3	5	12	<8	<5	13	2.7	9	2.5	2.1	N.d.	N.d.	N.d.	N.d.
Taken at Ardencaple Bay at $\frac{1}{2}$ m. on 27. vi. 50. Stored in Pyrex bottle	<0.3	1.5	12	<5	<5	11	5	6	1.6	<0.4	10,000	<1,000	<3,000	<3,000
Taken at Atlantic Bridge at $\frac{1}{2}$ m. on 27. vi. 50. Stored in Pyrex bottle	<0.3	1.5	16	<5	<5	21	7	6	1	<0.4	9,000	<1,000	<3,000	<3,000
Noddack (1940)*	0.1	0.5	0.5	5	3	14	—	—	—	0.15	—	—	3	4
Ernst & Hoermann (1936)*	—	0.1	0.3-0.7	—	—	—	0.2-0.3	—	—	—	—	—	—	—
Atkins (1936)*	—	—	—	—	—	<8	—	—	—	—	—	—	—	10
Haber (1928)*	—	—	—	—	—	—	—	—	—	0.3	—	—	—	—
Goldschmidt (1937)*	—	—	—	—	—	—	—	—	—	—	—	50	—	—

* Quoted from Harvey (1945).

TABLE VII. 'CONCENTRATION FACTOR', OR RATIO OF TRACE-ELEMENT CONTENT IN ALGAE (FRESH WEIGHT) TO TRACE-ELEMENT CONTENT IN SEA WATER

Sample	Ni	Mo	Zn	V	Ti	Cr	Sr
<i>Pelvetia canaliculata</i>	700	8	1,000	100	2,000	300	20
<i>Fucus spiralis</i>	1,000	15	—	300	10,000	300	8
<i>Ascophyllum nodosum</i>	600	14	1,400	100	1,000	500	16
<i>F. vesiculosus</i>	900	4	1,100	60	2,000	400	18
<i>F. serratus</i>	600	3	600	20	200	100	11
<i>Laminaria digitata</i> frond, Atlantic Bridge	200	2	400	10	90	200	90
<i>L. digitata</i> stipe, Atlantic Bridge	300	3	600	10	200	230	16
<i>L. digitata</i> frond, Ardencaple Bay	200	2	1,000	20	100	200	18
<i>L. digitata</i> stipe, Ardencaple Bay	400	2	900	30	90	200	14

ACKNOWLEDGEMENTS

This work forms part of a programme of research and development of the Scottish Seaweed Research Association, and the writers are indebted to the Director for permission to publish. The writers also wish to thank the staff of the Plymouth Marine Biological Laboratory, who collected the water samples off Plymouth, and the staff of the Macaulay Institute for Soil Research, Aberdeen, for the very valuable assistance in carrying out the spectrographic analyses.

SUMMARY

Some common Scottish Laminariaceae and Fucaceae have been analysed spectrographically, and seventeen of the minor elements determined.

The results show that there is a seasonal variation and considerable variation in the content of these elements in different species taken from the same habitat.

With the Laminariaceae the trace elements are more concentrated in the perennial stipe than in the attached frond, and are generally less than in the Fucaceae.

Samples of sea water taken off Plymouth and the West Coast of Scotland have also been analysed spectrographically and fourteen of the minor elements determined.

Concentration factors are given showing the extent to which marine algae can accumulate the trace elements. Values of over 1000-fold concentration are reported. *Fucus spiralis*, for example, contains 10,000 times more titanium than the surrounding sea water.

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