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Technical Report No. 50

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Office of Naval Research
Contract N8onr-520/III
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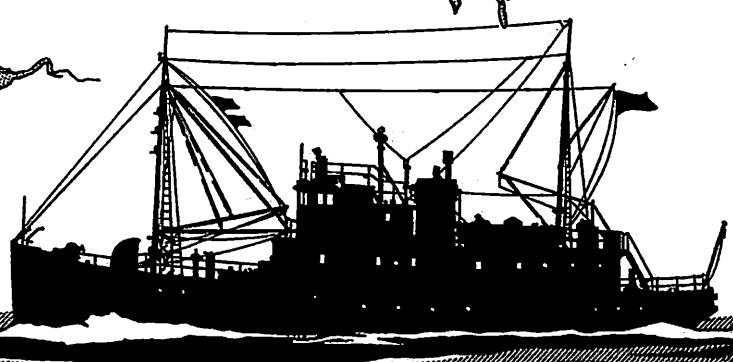

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ABSTRACT

The occurrence of nickel in sea water and certain materials of geologic and biologic origin has been reviewed. Methods for the analysis of nickel in sea water, in particulate material, and in marine organisms were investigated and modified. Nickel was satisfactorily recovered from sea water using sodium carbonate solution as the precipitant. The water should be treated immediately after sampling to minimize loss of nickel from solution through biological activity. In the preliminary treatment of marine organisms for nickel analysis, wet combustion was preferred to direct ashing as considerable nickel appears to be carried off with the smoke.

Sea water from Puget Sound and adjacent areas contain about 0.034 ug.-at. of nickel per kilogram in solution and 0.005 ug.-at. of nickel as particulate. River and lake waters contain slightly less soluble nickel than found in sea water. Plankton were found to contain twice as much nickel as the higher marine plants and ten times that found in the fish and mollusks. Indications are that plankton from the euphotic layers contained about twice the concentration found near the aphotic zone, and that nickel is probably concentrated by bacteria. Plants and animals concentrate nickel in their calcareous skeletons in the same degree as in soft tissues.

The amount of nickel in sediments collected across the continental shelf to the deep sea increased as the depth of water and distance from shore increased. Nickel is apparently halmyrolysed from shallow-water sediments and enriched in deep-sea deposits by several scavengers.

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The Determination and Occurrence of Nickel in Sea Water, Marine Organisms, and Sediments[1]).

By

Taivo Laevastu and **Thomas G. Thompson,**
Department of Oceanography, University of Washington, Seattle.

Introduction.

The objectives of this investigation were to (1) review the literature dealing with the occurrence of nickel in sea water and in marine organisms, and examine the methods previously used for the estimation of nickel in marine organisms, (2) devise a convenient and reliable method for the determination of minute quantities of nickel in natural waters, (3) determine the concentrations of nickel occurring in the waters of the Puget Sound region, and (4) investigate the occurrence of nickel in certain marine fish, plankton, and deposits of the Puget Sound region.

Previous Studies on the Occurrence of Nickel in Sea Water, Organisms, Sediments, and Rocks.

Relatively few analyses of nickel in sea water are given in the literature. The data available have been summarized in Table 1. The concentrations given range from 0·002 μg.-at. of nickel for the North Sea to 0·131 μg.-at. for Sevastopol Bay in the Black Sea. In most of the papers cited nothing is said about precautions taken to prevent contamination of the samples or corrections made for traces of nickel in the reagents utilized. The data available vary by more than one order of magnitude, and due to the meagreness of information it is impossible to state whether the differences are due to regional or seasonal variations, or to the sensitivities or errors in the methods employed for analysis.

[1]) Contribution No. 192 from Department of Oceanography, University of Washington, Seattle, Washington, U. S. A.

Table 1.

Amounts of nickel in water as reported by various investigators.

Place of collection	µg./Kg.	µg.-at./Kg.	Investigators and References
North Sea	0·12	·002	Ernst and Hörmann (1936)
Sea water (est.)	1·0	·017	Maljuga (1939)
Skagerak, Gullmarfjord	·5	·0085	I. and W. Noddack (1939)
Black Sea	6·0	·102	Maljuga (1942), according to Vinogradov (1944)
Sea water (est.)	3·0	·051	Vinogradov (1944)
Black Sea, 1700 m.	4·5	·076	Maljuga (1945)
Black Sea, Sevastopol Bay	7·7	·131	Maljuga (1945)
Black Sea, Karedag Biol. Stat.	5·1	·087	Maljuga (1945)
Barents Sea, off Yarnysh Bay	3·4	·058	Maljuga (1945)
Sea water, avg. (est.)	3·4	·058	Maljuga (1945)
20 miles off Plymouth, 20 m. depth, 22. II. 50	5·5	·093	Black and Mitchell (1952)
Ardencaple Bay, 0·5 m. depth, 25. VI. 50	1·5	·025	Black and Mitchell (1952)
Atlantic Bridge, 15 m. depth, 27. VI. 50	1·5	·025	Black and Mitchell (1952)
Pacific Ocean, off Shirahama, Wakayama, 20. X. 50	0·75	·013	Ishibashi (1953)
Average:	3·4	·053	
River waters, average	4·8	·082	Maljuga (1945)
Lake waters, average	17	·29	Maljuga (1945)

The amount of nickel reported in materials of geologic or biologic origin by other investigators is given in Tables 2 and 3. These data show marked variation in the trace quantities of nickel in different rock formations. Besides meteorites and ore formations the basic igneous rocks, red clays and organic sediments are the richest in nickel, whereas acid igneous rocks, sedimentary rocks, soils and shallow water sediments have the lowest nickel content. Some oolitic iron ores are relatively high in nickel.

There are marked differences in the quantities of nickel in plants and animals. I. and W. Noddack (1939) state that the element must be essential for animal life because one kilogramme of dried organisms contained 41,000 times the amount of nickel contained in one kilogramme of sea water. They stated also that the concentration of cobalt in marine animals is about one-tenth that of the nickel concentration. Black and Mitchell (1952), employing similar reasoning, found a concentration factor of 540 for nickel in sea plants as compared to sea water. The concentration of cobalt was equivalent to about one-fourth that of the nickel concentration of the plants. They also noted that the nickel concentrations were higher in spore-forming *Laminaria* than in those not forming spores, and that greater quantities of nickel occur in the plants in the summer than in the winter months. On the other hand, Bertrand and Mâcheboeuf (1925) claimed that animals have far less nickel than cobalt in their tissues. Kent and

Table 2.

Amounts of nickel in rocks, soils, and sediments as reported by various investigators.

Element	Ni in mg./Kg.	Ni in mg.-at./Kg.	Investigators and References
Basic igneous rocks	65	1·1	Lundegårdh (1949)
	100—2,000	1·7—34·0	Vogt (1923)
Acid igneous rocks	tr.—5	tr.—·085	Vogt (1923)
	8	·14	Lundegårdh (1949)
Igneous rocks	80	1·4	Sandell, Goldich (1943)
	200	3·4	Clarke (1924)
	48	·82	Lundegårdh (1949)
	100	1·7	Goldsmidt (1937)
Oolitic iron ores	—39,000	—664	Maljuga (1939)
Sedimentary rocks	tr.—·28	tr.—·0048	Maljuga (1939)
Sandstones	2—8	·034—·14	Rankama, Sahama (1949)
Shales	24	·41	Rankama, Sahama (1949)
Limestones	0	0	Rankama, Sahama (1949)
Mineral soils	20	·34	Goldsmidt (1954)
Soils	8·3—730	·14—12·5	Maljuga (1939)
Meteorites	16,000	272	Rankama, Sahama (1949)
Asphalt	3,000	51	Wells (1946)
Naphtha	250—1	4·25—·017	DeGolyer (1924) quoted by Maljuga (1939)
Coal	220—5·2	3·7—·089	Kraut (1906)
Coal ash	700	12	Goldsmidt (1954)
Oak humus ash	100	1·7	Goldsmidt (1954)
Red clay (composite)	253	4·31	Clarke (1924)
Red clay (composite)	259	4·41	Young (1954)
Red clay, Pacific Ocean	164	2·79	Landergren (communication)
Terrigenous clays	498	8·49	Clarke (1924)
Sea deposits	250	4·25	Maljuga (1945)
Delta sediments	110	1·87	Maljuga (1945)
Near-shore sediments (composite)	47	0·8	Young (1954)
Manganese concretions	7,700	131	Irvine and Gibson, quoted by Maljuga (1945)

McCance (quoted by Monier-Williams, 1949) found that in humans much of the nickel consumed is discharged in the urine. Stiles (1946) states that nickel is more frequent in animals than cobalt, that nickel is irregularly distributed, and that little is known of its presence in various organs. He indicates that the availability of nickel to plants increases the growth but that there is no evidence that the element is essential. Wallace (1952) has demonstrated that excess nickel is toxic to plants and organisms and that its presence in excess may induce an iron deficiency. Vinogradov (1953) states that Paulais (1936) has found a concentration of nickel in the gills and liver of molluscs.

Goldsmidt (1954) assumed that divalent nickel accompanies divalent magnesium, cobalt, and iron because of the similarity of the ionic radii.

Table 3.

Amounts of nickel in plants and animals as reported by various investigators.

Element	Ni mg./Kg. Dry matter	Ni mg.-at./Kg. Dry matter	Investigators and References .
Seaweeds	3·7	·063	Black and Mitchell (1952)
	·099—·83	·0017—·014	Maljuga (1939)
Marine plants	·5	·0085	Maljuga (1939)
Freshwater algae	·0017—·012	·00003—·0002	Maljuga (1939)
Fruits and grains	·01—2·0	·00017—·034	Maljuga (1939)
	·9	·015	Bertrand and Mokragnatz (1930)
Lettuce, cabbage, spinach, and peas	1·5—3·0	·026—·051	Bertrand and Mokragnatz (1930)
Pastures	·56—3·65	·01—·062	Scott and Mitchell (1943)
Whole plants on living basis	·013—1·5	·00022—·026	Maljuga (1939)
Calanus finmarchicus	1·2	·0204	Maljuga (1939)
Fish	·015	·00026	Bertrand and Mâcheboeuf (1925)
Osmerus eperlanus	·68	·0116	Bertrand and Mâcheboeuf (1925)
Gadus sp.	1·1	·0187	Maljuga (1945)
Polychaetes and molluscs	8—100	·14—1·7	Fox and Ramage (1931)
Mytilus edulis (meat)	2·35	·040	Bertrand and Mâcheboeuf (1925)
Sponges	1·8—6·14	·036—10·45	Bowen and Sutton (1951)
Marine animals	20·6	·35	I. and W. Noddack (1939)
	·5	·0085	Maljuga (1939)
	·01—20	·00017—·34	Maljuga (1939)
Land animals	·01—·05	·00017—·00085	Maljuga (1939)
Human liver	·09	·0015	Bertrand and Mâcheboeuf (1925)
Living matter	·01—20	·00017—·34	Vinogradov (1937)

The Determination of Nickel in Sea Water.

Ernst and Hörmann (1936) isolated nickel from the major constituents of sea water by adding a solution of a ferrous salt and then precipitating the ferrous and nickel sulphides with ammonium sulphide. Ishibashi *et al.* (1951) employed a ferric salt solution to serve as a collector, precipitating the traces of nickel with the ferric hydroxide by addition of ammonium hydroxide. Samples of 40 to 60 litres were taken for each analysis. Several other investigators used spectroscopic methods, concentrating samples of water by evaporation.

In the present investigation, the addition of a collector was unnecessary, because in an alkaline solution the traces of nickel were carried down with the precipitates of magnesium hydroxide or carbonate. The first reagent employed was sodium hydroxide, but this proved unsatisfactory because the analytical grade chemical contained as much as one microgramme of nickel per gramme. Sodium carbonate was obtainable practically free of nickel, and this was the reagent used. For the final estimation of nickel the procedure given by Sandell (1950) was employed. The complete procedure is as follows:—

Collection of samples.

Extreme care was exercised in the collection of samples to prevent contamination. The water-sampling bottle described by Thompson and Chow (1955) insured the collection of samples at various depths free of contamination. Samples were treated for analysis immediately after collection. If this were not done, micro-organisms in the water would continue to grow and take up nickel from solution.

Chemicals.

All chemicals should be completely free of traces of nickel or blank determinations made to ascertain the quantities of nickel present.

Sodium carbonate solution:	50 g. of reagent grade anhydrous sodium carbonate dissolved in 1 litre of distilled water.
Hydrochloric acid:	Solutions of 6N and 0·6N prepared by dilution.
Ammonium hydroxide:	Concentrated reagent grade.
Dimethylglyoxime:	A 1 % solution is prepared by dissolving in ethyl alcohol.
Bromine water:	Saturated solution.
Sodium citrate solution:	A 10 % solution prepared by dissolving the salt in water.
Chloroform:	Reagent grade.

Preparation of standard nickel solutions.

Weigh out exactly 0·3365 g. of nickel ammonium sulphate hexahydrate crystals or 0·0500 g. of pure nickel wire. Dissolve the salt in redistilled water or the wire in dilute nitric acid, transfer quantitatively to a volumetric flask, and dilute to one litre. Ten ml. of this solution is taken and diluted to exactly one litre. One ml. of the latter solution is equivalent to 0·5 microgrammes of nickel (0·0085 μg.-at./ml.).

Pure nickel wire is the most desirable substance to employ for preparation of standard solutions. Other standard solutions were prepared with the hexahydrates of nickel chloride, nickel sulphate, and nickel ammonium sulphate. The results obtained from the different standards are illustrated in Figure 1 and show that of the hydrates, if it is necessary to use one as a standard, nickel ammonium sulphate is preferable.

Procedure.

Two-litre samples of sea water are filtered through a type HA millipore filter. The filtrate is assumed to contain all the nickel in true solution (Lewis and Goldberg, 1954), whereas the detritus retained on the filter may be taken and analysed for nickel, as described later, and reported as particulate nickel.

One hundred ml. of sodium carbonate solution is added to the

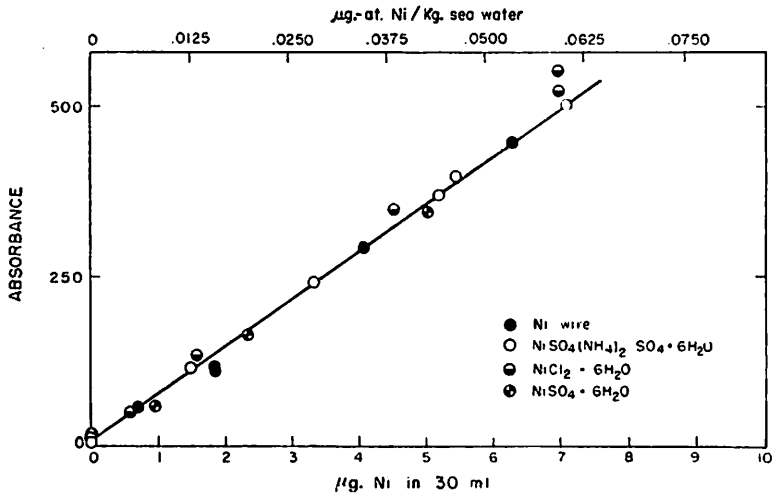

Figure 1. Nickel calibration graph.

filtrate which is then allowed to stand for about 12 hours. The supernatant liquor is syphoned through a millipore filter, and then the bulk of the precipitate is transferred to the filter. The residual precipitate is dissolved in a small quantity of 6N hydrochloric acid, the solution transferred to a small beaker to which the bulk of the precipitate retained by the filter has been introduced. The filter is then washed quantitatively with 6N hydrochloric acid and the washings combined with the solution resulting from dissolving of the precipitate. The total volume should not exceed 25 ml. and should be kept constant. This solution, after the addition of 15 ml. sodium citrate solution is made ammoniacal to a pH 8. It is then cooled rapidly, transferred to a separatory funnel, 2 ml. dimethylglyoxime added, and then shaken with 3 ml. chloroform for about half a minute. The chloroform extracts the nickel dimethylglyoxime. It is drawn off into another separatory funnel, care being taken not to permit any of a possible slight precipitate that may have formed over the chloroform to pass into the funnel. This process of extraction is repeated twice. The combined extractions are then shaken with 5 ml. 1 : 50 ammonium hydroxide and the chloroform drained into another separatory funnel. The ammonium hydroxide is shaken with 2 ml. chloroform and the latter drained and combined with the previous chloroform solution. This solution is then shaken with 5 ml. 0·6N hydrochloric acid, the nickel dissolving in the acid and the dimethylglyoxime being retained by the chloroform. The process is repeated. The combined solutions are then transferred to a flask which will permit eventual dilution to a volume of exactly 30 ml. Ten drops of bromine water are added, and after a few minutes ammonium hydroxide is slowly introduced until the bromine colour disappears. When this occurs, 0·5 ml. ammonium hydroxide is added in excess, followed by the addition of one ml. dimethylglyoxime and the

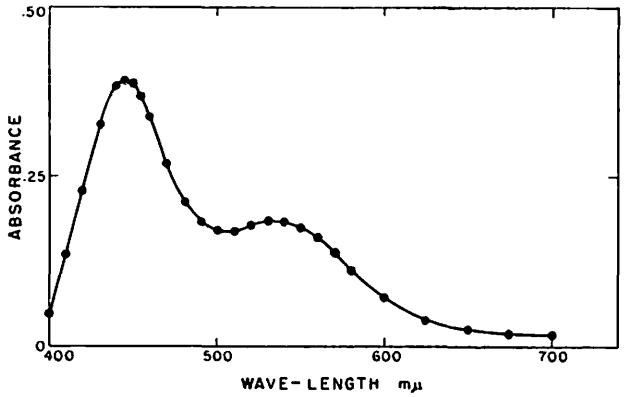

Figure 2. Absorbance of nickelic dimethylglyoxime complex.

solution made up to a volume of exactly 30 ml. It is then transferred to a 100 mm. cell and its absorbancy determined with a Beckman spectrophotometer. The absorbancy of the solution is measured at 450 mμ. The full colour intensity develops almost immediately upon the addition of dimethylglyoxime. The measurement of the absorbancy is made 15 minutes after the addition of this reagent and transference to the cell.

Preparation of calibration graph.

Varying quantities of the standard nickel solution are pipetted into separatory funnels so that a range of from 0·5 to 10 microgrammes (0·0085 —0·170 microgramme atoms) of nickel are in six solutions. A volume of hydrochloric acid, equivalent to that used in dissolving the precipitated carbonates as described above, is added to each solution. Fifteen ml. of sodium citrate is introduced and each of the standard samples is treated in the same manner as given in the procedure, using the identical reagents in the same quantities. The absorbancies obtained are plotted against the concentration of nickel. From this graph (Figure 1) and from the absorbancies obtained on the water samples being analysed, the actual concentration of nickel may be ascertained. Figure 2 shows the absorbancy plotted against the wave-length.

By treatment of the standards in the manner described, there was compensation for errors occurring during the extraction processes and also for traces of impurities that might have been present in the reagents used.

Comparison and validity of procedures.

The method of Ishibashi *et al.* (1951) was questioned, as complete retention of nickel by the ferric hydroxide precipitate might not occur because of the exceedingly small concentration of nickel in sea water and the tendency to form soluble nickel ammonia ions. A comparison of the Ishibashi procedure with the method proposed was made, using, however, only two litres of water instead of 60 litres

required by Ishibashi. A sample of sea water was analysed with ferric hydroxide used as the collector in an ammoniacal solution. Nickel was also determined on a similar two-litre sample by precipitating the collectors already present in the water with sodium carbonate. In each case check determinations were made and the following results were obtained:—

	Microgramme-atoms nickel per Kg. of sea water		
	Portion A	Portion B	Average
Method of Ishibashi *et al.*	·015	·017	·016
Sodium carbonate method	·0255	·024	·025

These results demonstrate that greater quantities of nickel were precipitated when the natural collectors and sodium carbonate were used than when ferric hydroxide in ammoniacal solution was employed.

When a solution containing nickel is made ammoniacal, the very soluble nickel ammonia ion tends to form. However, in the presence of ferrous or ferric hydroxides most of the nickel will be retained by the precipitates, but due to the exceedingly small concentration of nickel occurring in sea water the precipitation is not quantitative.

To test further the validity of the sodium carbonate procedure, it was first deemed desirable to prepare a synthetic sea water, free of nickel, and to which known concentrations of nickel could be added. However, the synthetic sea water prepared contained nickel (present as an impurity in the chemicals used) in quantities far greater than the natural sea water and the concentrations of nickel proposed for addition, even though reagent grade chemicals were employed. Therefore, several different samples of natural sea water were analysed for nickel, varying concentrations of known amounts of nickel were then added to other samples of the same waters, and the total nickel was determined. The results were as follows:—

Ni in 2 litres sea water μg.-at.	Ni added μg.-at.	Ni recovered μg.-at.	Percentage recovered
·055	·085	·123	80
·055	·1365	·169	83
·055	·085	·143	103
·055	·085	·136	95
·0485	·085	·146	115
·053	·0425	·095	99
		Mean recovery:	96

Influence of storage on the nickel content of water.

It is well known that storage of samples of sea water before analysis markedly affects the concentration of some of the nutrient salts. It was found that the concentration of nickel was affected similarly.

Large samples of water were collected and divided in five portions. One portion was filtered and analysed immediately for nickel. Other portions were stored in darkness or subjected to the action of artificial illumination for a period of two to three weeks before filtration and

analysis. To another portion hydrochloric acid was added before storage and to still another carbon disulphide. The final analyses of these various portions yielded the following results:—

	Sample I	Sample II
	μg.-at. Ni/Kg.	
Immediate filtration and analysis	0·036	0·031
Storage in darkness for 3 weeks	·010	
Storage in darkness for 2 weeks		·028
Storage in light for 3 weeks	·026	
Storage in light for 2 weeks		·024
Addition of 7·5 ml. 12N HCl per litre before storage. Stored for 3 weeks	·041	
Stored for 2 weeks		·045
Addition of several drops of CS_2 before storage. Stored for 3 weeks	·016	
Stored for 2 weeks		·022

These results demonstrate that nickel is removed from solution by biological action on storage. The addition of hydrochloric acid halted biological action but caused some of the particulate nickel to go into solution, whereas carbon disulphide may have caused precipitation of some of the nickel. Thus filtration and analysis immediately after collection of samples is essential if one wishes to differentiate between the nickel actually in solution and particulate nickel. If one is interested solely in the total nickel content of a sample, then immediately after collection, two litres of the sample should be measured, transferred to an appropriate container, and 100 ml. of sodium carbonate solution added. Continuation of the analysis may then proceed according to convenience.

Determination of Nickel in Marine Organisms.

Two methods for the preliminary treatment of various samples of biological origin prior to the determination of nickel were studied, namely, direct ashing and wet combustion. The results, with the two methods used for destruction of organic matter, as evidenced by the amount of nickel actually determined, are given in Table 4. In all materials, with the exception of plankton, abnormally low results were secured on samples that had been ashed. Due to the high mineral content of plankton (as indicated by the voluminous and porous residue obtained on ignition) there were only slight differences observed in the results obtained by the two methods.

When wet combustion was deemed advantageous, the following procedure was used:— 2 to 8 g. of oven-dried material were weighed in an Erlenmeyer flask, treated with nickel-free concentrated sulphuric acid, and heat applied until the mass was colourless or yellow-brown. When all organic matter had been destroyed and much of the excess sulphuric acid fumed off, the residue was cooled and then treated with 10 ml. 6N hydrochloric acid. The solution was diluted to about 25 ml., heated again, and any residue was removed by filtration, the filtrate

Table 4.

Comparison between ashing and wet combustion of biological materials.

	Ni in mg.-at./Kg. dry matter	
	Wet combustion	Ashing
Plankton	·104	·101
Mackerel meat	·005	·0035
Perch meat	·007	·0035
Kelp	·012	·007
Dorsal skin from flounder	·017	·0085

being received in a separatory funnel. Fifteen ml. of sodium citrate solution was then added, and the analysis for nickel was continued as described above. The sulphuric acid used for wet combustion should be analysed for nickel by performing several blank determinations, and the correction applied.

Analytical Results.

Nickel in solution in sea water.

In Table 5 are given the results obtained on samples of filtered and unfiltered sea water collected in Puget Sound and adjacent areas. The mean value found for filtered sea water was ·034 microgramme atoms of nickel per kilogramme of sea water; extreme values varied from 0·024 microgramme atoms for a water collected at the surface in Hood Canal in May 1954 to 0·046 microgramme atoms for a sample secured from 35 metres in Puget Sound off Golden Gardens in October 1954. These data are more than one order of magnitude higher than the values given by Ernst and Hörmann (1936) and twice as great as those cited by Ishibashi (1953) but are in agreement with the concentrations given by Black and Mitchell (1952).

From the data given in Table 5, it appears that the concentration of nickel in the surface waters is less than that obtained in waters of the depths. A seasonal variation in the concentration of nickel, and that nickel is not a limiting factor in organic production, are also indicated.

Interstitial water.

Water was collected from holes dug in several mud flats during low tide. The nickel content, as indicated from data presented in Table 5, was slightly higher than that of the adjacent waters.

River and lake water.

Samples of water taken from Lake Washington and two rivers, the Hoh and Sol Duc, were analysed for nickel, and the data given in Table 5 show that the nickel content of these fresh waters was slightly lower than that of sea water.

Table 5.

Nickel content of waters in Puget Sound and adjacent areas.

Date of collection	Place of collection	Depth m.	Cl ‰	Soluble Ni µg.-at./Kg.
1954				
16. April	San Juan Archipelago, off McConnell Island	0	16·3	0·027
22. May	Hood Canal, off Misery Point	0	14·0	·024
		100	16·3	·027
7. July	San Juan Archipelago, Friday Harbor	0	16·2	·029
12. October	Puget Sound, off Golden Gardens, Seattle	0	14·9	·036
		35	16·6	·046
		80	16·6	·037
21. October		35—40	16·5	·044
3. November	Puget Sound, off West Point	0	16·4	·027
		75	16·7	·032
1955		150	16·8	·046
7. January	Puget Sound, off Point No Point	0	15·9	·030
				Total Ni µg.-at./Kg.
5. January	47°16′N., 127°04′W	0	17·82	0·048
		50	17·83	·041
		100	18·55	·054
		200	18·66	·034
6. January	Strait of Juan de Fuca, off Race Rocks	0	17·30	·034
1954	Fresh Waters			Soluble Ni µg.-at./Kg.
21. October	Lake Washington	0	0	0·026
20. December	Hoh River, Olympic Peninsula		0	·027
	Sol Duc River, Olympic Peninsula		0	·029
	Interstitial Waters from Mud Flats			
22. May	Head of Hood Canal			
	Water from surface of mud		9·0	0·031
	Hole dug at low tide mark		6·0	·039
	Brown Island, Friday Harbor			
	Hole dug in mud at high tide mark		16·3	·039
	Hole dug in mud at low tide mark		16·2	·024

Particulate nickel.

The residues retained by the millipore filters were analysed for nickel and the quantities reported as particulate nickel per kilogramme of water. The results obtained on detritus are shown in Table 6. The amount of particulate nickel averaged about 0·005 microgramme atoms per kilogramme of water, or about 15 % of the concentration of the soluble nickel.

Table 6.

Particulate nickel in water from Puget Sound and adjoining areas.

Date of collection	Place of collection	Depth m.	Ni μg.-at./Kg. of sea water
1954			
16. April	San Juan Archipelago, off McConnell Island	0	0·007
12. October	Puget Sound, off Golden Gardens, Seattle	0	·003
		35	·007
		80	·003
21. October		35—40	·003
3. November	Puget Sound, off West Point	0	·005
		75	·007
		150	·007
21. October	Fresh water from Lake Washington	0	·005
20. December	Hoh River, Olympic Peninsula		·154
	Sol Duc River, Olympic Peninsula		·021

The particulate nickel in the waters of the two rivers investigated was considerably higher than that found in sea water, as shown by the data in Table 5. The Hoh River water contained much finely divided clay in suspension and the particulate nickel was much higher than that of the Sol Duc. The nickel present was of mineral origin.

The relatively high concentration of nickel in the particulate matter, as compared with the soluble nickel, gives an explanation of the very high values reported by Maljuga (1939, 1945) for river and lake water, as this investigator did not distinguish between soluble and particulate nickel.

Nickel in marine plankton.

The concentration of nickel in marine plankton is given in Table 7. The plankton was collected with metal-free nets, the major components determined under a binocular microscope, and the abundance reported as percentage by volume. The plankton samples were ashed before analyses for nickel were made.

Fleming (1940) reported that in general one gramme of dry plankton is equivalent to 16 g. of fresh plankton. In accumulating the data given in Table 7, this statement was substantiated; but, as would be expected, there is a deviation from each particular sample depending upon its biological composition.

From the data presented, it appears that plankton collected in the spring months contains more nickel than that secured in the autumn. The nickel content of plankton collected at or near the surface showed a concentration of nickel twice that of samples obtained in deeper water. The concentration factor of nickel in fresh plankton in relation to the concentration of nickel in sea water was found to vary from 170 : 1 to 970 : 1 with an average value about 500 : 1. The corresponding concentration factor for phosphates is about 20,000 : 1. This shows that plankton organisms are highly selective as to intake of ions.

Table 7.

Nickel content of marine plankton.

Date of collection	Place of collection	Depth m.	% dry matter	Constituents by volume %	Ni mg.-at./Kg. dry matter	
1954						
24. April	Puget Sound, off Golden Gardens, Seattle	0	8·4	*Pseudocalanus*	85	·525
				Acartia	10	
		50—75	8·1	*Pseudocalanus*	70	·262
				Sagitta	25	
23. May	Hood Canal, off Misery Point	0	12·5	*Peridinium*	60	·213
				Coscinodiscus	28	
				Pseudocalanus	8	
		70	9·6	*Coscinodiscus*	60	·136
				Pseudocalanus	30	
2. July	San Juan Archipelago, East Sound			*Schröderiella* and *Chaetoceras*	85	·174
		0	4·8	Other diatoms	13	
		10	6·0	*Schröderiella* and other diatoms	70	·094
				Ceratium	30	
15. October	Puget Sound, off Golden Gardens, Seattle	1	—	*Coscinodiscus*	90	·153
		40	—	*Coscinodiscus*	90	·101

In the Annual Report of the Institute for Seaweed Research (1953) it is stated that cobalt is assimilated by seaweeds during the processes of photosynthesis and emitted during the hours of darkness. As nickel is analogous to cobalt a preliminary test was performed in order to ascertain whether a similar behaviour could be noted, and thus give a possible explanation of the higher nickel content of surface plankton. Samples of sea water were taken, enriched by the addition of nickel, and living plankton was added. One bottle was placed in darkness for 30 hours and the other exposed to artificial light for a similar length of time. On filtration and the analysis of the waters, the following results were obtained:—

	Sea water Ni μg.-at./Kg.	Plankton and bacteria Ni μg.-at./g. dried material
Original nickel-enriched water	0·073	0·078
After 30 hours exposure to light with plankton	·025	·204
After 30 hours in darkness with plankton	·027	·150

These results demonstrate that besides the intake of nickel by photosynthetic activities, the decrease in concentration of nickel in the water may be attributed to selective adsorption or absorption or to assimilation by bacteria. It was noted that there was a decided growth of bacteria in the bottles after standing 30 hours. Bowen and Sutton (1951)

Table 8.
Nickel in fish.

	Length in cm.	Age years	% Dry matter	Ni in μg.-at./Kg. dry matter
Fish				
Phanerodon tureatus (flesh)	32·5	7+	15·4	7·0
Phanerodon tureatus (flesh)	23·0	3+	21·7	6·0
Taeniotoca lateralis (flesh)	20·0	2+	19·6	7·0
Pneumatophorus diego (flesh)	24·0		28·6	5·5
Pneumatophorus diego (flesh)	33·0		28·6	5·5
Platichtys stellatus (flesh)	29·5	5+		9·2
Platichtys stellatus (dorsal skin)				17·0
Oncorhynchus kisutch (flesh)	58·0	2+		28·9
Oncorhynchus kisutch (sperm)				20·3
Oncorhynchus kisutch (blood)				13·5
Oncorhynchus kisutch (liver)				8·5
Sardinops caerulea				5·1
Molluscs				
Siliqua patula (flesh)				12·6

Most of the above specimens were collected and identified by Dr. Lauren R. Donaldson and Dr. Allan C. DeLacy, of the School of Fisheries, University of Washington, and John S. Thompson, of the Washington State Department of Fisheries.

assumed that bacteria associated with marine sponges could concentrate nickel in much greater quantities than the sponges free of bacteria. Ericson and Lewis (1953) made a similar assumption for the assimilation of cobalt by bacteria attached to seaweeds.

Nickel in marine plants.

Some plants were collected in the late spring and early summer of 1954 and prepared for analysis by ashing of the material. No detailed data are here presented because of the unreliability of the analyses due to ashing. The mean concentration of nickel in the plants examined by this method was 85 microgramme atoms per kilogramme of dried material; but as shown in Table 4, this average figure is low because of ashing.

Nickel in fish.

Analyses for nickel on several species of fish were made, and the data are given in Table 8. The flesh of *Oncorhynchus kisutch* contained about three times the concentration of nickel as found in that of the other fish examined, and about twice that of the molluscs analysed. The nickel content found in the flesh of the fish examined, with the exception of *Oncorhynchus kisutch*, is the same order of magnitude as that reported by Maljuga (1939) for marine animals.

Nickel in calcareous skeletons of plants and animals.

As Rankama and Sahama (1949) report the absence of nickel in limestone, it was deemed desirable to ascertain the absence of the

Table 9.

Nickel in calcareous skeletons of plants and animals.

	Place of collection	Ni μg.-at./Kg.
Plants		
Coralliniaceae		
(*Lithothamnion* and other genera)	Off Sarasota, Florida	70
Animals		
Macoma nubilus	Puget Sound	5·3
Pecten diegensis	Puget Sound	7·8
Mytilus californiensis	Puget Sound	6·0

metal in biogen calcareous remains. A few analyses were made on recent clam shells and calcareous algae. The results are reported in Table 9 and show the same tenfold difference as given above for the nickel content of plants and animals. From this limited data, it might be concluded that plants and animals concentrate nickel in the calcareous skeletons in the same degree as in their organic tissues.

Nickel in marine sedimentary deposits.

Samples of marine sediments from different areas were examined for their nickel content, and the results secured are shown in Table 10.

Two of the samples analysed were shallow-water sediments from the coast of Florida and were supplied by Dr. Howard G o u l d. The composition as well as the nickel content of these samples are comparable to sandstone. The sample from Charlotte Harbor contained more nickel than that from Tampa Bay and was also richer in organic matter, which may possibly explain the higher nickel content.

The sediment samples from Puget Sound and the adjacent Strait of Juan de Fuca and San Juan Archipelago are comparable, because of their composition, to the shales (Table 2) but have a higher nickel content. This high nickel content may be attributed to organic matter and to the adsorption of nickel ions by clay particles. The source of nickel, aside from that occurring in the inflowing waters, may be the result of freshwater runoff and the weathering and halmirolysis of rocks. This influx of nickel is removed from the surface layers by organisms or by adsorption by clay particles. A portion of these nickel scavengers eventually become sedimentized.

A series of sediment samples was collected on a course from near the entrance of the Strait of Juan de Fuca, across the continental shelf to the deep water of the Pacific. These results, given in Table 10, demonstrate that the nickel content of the sediments appreciably increases seaward. It may be assumed that nickel is halmirolysed from shallow-water sediments (Y o u n g, 1954) and enriched in deep-sea deposits by scavenging manganic hydroxide (G o l d b e r g, 1954). As indicated above, organic matter, clay minerals, and iron hydroxide may be as effective scavengers as manganese hydroxide. Further evidence of the effectiveness of iron as a scavenger is indicated by the high nickel content of marine oolitic iron ores. From the data given in Table 9 it

Table 10.

Nickel content of marine sediments.

Place of collection	Depth m.	Description of sediment	Ni mg.-at./Kg.
Florida			
Tampa Bay	3	Quartz sand	0·024
Charlotte Harbor	3·5	Sandy mud	·082
Puget Sound Area			
Carr Inlet, Still Harbor	112	Silty clay and mud	·62
Hood Canal	148	Silty clay and mud	·66
Jefferson Head	278	Silty clay and mud	·75
San Juan Archipelago			
East Sound, Orcas Island	25	Silty organic mud	·57
San Juan Canal	75	Silty clay and mud	·62
Strait of Juan de Fuca			
48°13′N., 122°53′W.	46	Gravel and organic matter	·95
48°14′N., 123°24′W.	127	Sandy mud	·51
48°17′N., 124°02′W.	188	Sand	·36
North-east Pacific Ocean, Continental Shelf			
47°45′N., 125°02′W.	141	Sand and silt	·54
47°46′N., 125°05′W.	208	Sandy silt	·53
48°06′N., 125°10′W.	456	Silt and clay	·63
47°55′N., 125°30′W.	640	Clay	·87
47°50′N., 125°35′W.	841	Clay	·94
47°50′N., 125°43′W.	1180	Clay	·81
47°50′N., 125°48′W.	1490	Clay	1·05
47°54′N., 126°06′W.	1848	Clay	1·10
Gulf of Alaska			
53°15′N., 149°20′W.	4390	Diatom ooze 2—4 cm. below sediment surface	1·82
54°08′N., 141°15′W.	3620	Diatom ooze with red clay 2—4 cm. from sediment surface	2·90
54°08′N., 141°15′W.	3620	Foram ooze 110—112 cm. from sediment surface	·66
56°14′N., 152°58′W.	970	Terrigenous clay 55—57 cm. below sediment surface	·68
Bering and Chuckchee Seas			
74°14′N., 162°47′W.	30		·13
70°55′N., 158°67′W.	25		·17
68°19′N., 166°41′W.	20		·45
66°12′N., 162°40′W.	14		·51

appears that the formation of calcareous material also may be considered as a scavenger of nickel. Such being the case, the nickel in the calcareous debris will be subjected to halmirolysis.

In such a complex system as exists on or near the sea floor, halmirolysed nickel will tend to be reprecipitated or re-adsorbed by substances giving a more stable and less soluble form. Thus calcium

carbonate is subjected to a slow re-arrangement because of the chemical nature of the carbonate-bicarbonate-carbonic acid system. Some of the nickel liberated will be scavenged by the iron or manganese compounds, becoming very insoluble because of the characteristics of the iron or manganese compounds and the basic nature of the system.

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Summary.

Methods for the analysis of nickel in sea water, in particulate material, and in marine organisms were investigated and modified. By using collectors already present in sea water and sodium carbonate solution as the precipitant, a more satisfactory recovery of nickel was obtained. In the preliminary treatment of marine organisms for nickel analysis, wet combustion was preferred to direct ashing as a relatively large proportion of nickel appears to be carried off with the smoke. As nickel may be removed from solution by biological activity, samples of water should be treated immediately after sampling. Sea water from Puget Sound and adjacent areas contain about 0·034 μg.-at. of nickel per kilogramme in solution and 0·005 μg.-at. of nickel as particulate. River and lake waters contain slightly less soluble nickel than is found in sea water. The plankton examined contained twice as much nickel as the higher marine plants and more than ten times the amount found in the fish and molluscs examined. Indications are that plankton collected in the euphotic layers contained about twice the concentration found near the aphotic zone, and that nickel is probably concentrated by bacteria. Plants and animals concentrate nickel in their calcareous skeletons in the same degree as in soft organic tissues.

The amount of nickel in the sediments collected across the continental shelf to the deep sea increased as the depth of water and distance from shore increased.

Nickel is halmirolysed from shallow-water sediments and enriched in deep-sea deposits by several scavengers.

[1]) Contract No. N80NR-520/III, Project NR083-012.

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