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INTERCALIBRATION OF ANALYTICAL METHODS ON MARINE ENVIRONMENTAL SAMPLES

Prepared by:



INTERNATIONAL ATOMIC ENERGY AGENCY

C O N T E N T S

	<u>Pages</u>
1. Progress Report No. 13, June 1976	1 - 26
2. Progress Report No. 15, June 1977	27 - 47
3. Progress Report No. 16, March 1978	48 - 58
4. Progress Report No. 18, Oct. 1978	59 - 72
5. Progress Report No. 19, Nov. 1978	73 - 97
6. Progress Report No. 20, April 1980	98 - 106
7. Progress Report No. 1	107 - 131
8. Progress Report No. 2, April 1980	131 - 141

PROGRESS REPORT 3
INTERCALIBRATION OF LYTHAM THERMOCALIBRATION
NINE ENVIRONMENTAL SAMPLES

International Laboratory of Marine Activities
Municipality of Monaco
June 1976

Trace element measurements Oyster Homogenate (MA-M-1)

During the last few years several intercalibration exercises of nuclide measurements on marine environmental samples have been carried out by the Monaco Laboratory. Under the financial support of UNEP a new programme of intercalibration in trace element analysis of marine environmental samples was started in 1975. As a first step, in the framework of this new programme, an intercalibration exercise on an oyster homogenate MA-M-1 was carried out during the period of 1975-1976.

Approximately 2000 kg gross weight of oysters were collected in the vicinity of Sète, France, the soft parts (140 kg) were dissected and freeze-dried, powdered, sieved and homogenized. After testing the homogeneity, the sample was divided and packed into glass bottles of 30 g-capacity and these were sent to 113 laboratories. To date the results of 67 laboratories have been received. A list of the laboratories participating in the present intercalibration exercise is given as an Annex.

1. Results reported

Although the determination, detection or attempts at detection were made for more than 50 elements, only the results reported on 14 selected trace elements, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Ag, Cd, Sb, Hg and Pb are compiled in Tables 1, 2 and 3. Table 1 includes the results of Cr, Mn, Fe, Co, Ni, Table 2 those of Cu, Zn, As, Se, Ag, and Table 3 those of Cd, Sb, Hg and Pb. In each table abbreviated descriptions of the analytical methods used are also given. Although the results of 67 laboratories have been received to date, the results of only 60 laboratories had been compiled in this preliminary report. In compiling the data the following procedures were adopted:

- a) All values given in Tables 1, 2 and 3 were taken from the original reports of the participants with as little change as possible; significant figures of the values given in the tables are identical to those of the data reported by the participants.

- b) In cases where the data had apparently been reported on the freeze-dried weight basis, these were normalized to those based on the oven-dried weight at 105-110°C, using a factor measured at Monaco immediately before the dispatch.
- c) In cases where laboratories had not given laboratory averages for their individual measurements, arithmetical averages of the individual data were calculated and presented in the tables.
- d) The associated errors of the measurements were reported either in absolute values or in percentages. The percent errors are given in parentheses in the tables.
- e) In cases where laboratory averages were calculated at Monaco, associated errors for these averages were computed on the basis of the errors given for individual data when two or three data had been reported for one element; where four or more data were available for calculating the average, standard errors were computed from the individual data reported.

Although the compilation of results were made only on the 14 selected elements, results were reported on many other elements. The concentrations or presence of the following additional 29 elements were reported at least by one laboratory:

Li, B, F, Na, Mg, Al, Si, S, Cl, K, Ca, Sc, Ti, V, Br, Rb, Sr, Mo, Sn, I, Cs, Ba, La, Ce, Sm, W, Au, Th, U.

The following elements were reported as "not detected" or as less than the limits of determination for the methods employed:

Be, Ga, Ge, Zr, In, Eu, Tb, Lu, Hf, Ta, Bi.

The compilation of the results reported for those elements, not given in the present report, will be presented later.

2. Summary of the data

Tables 4, 5 and 6 show ranges and preliminary overall averages of the results reported by the 60 laboratories. In order to reject outlying data, Chauvenet's and Dixon's tests were employed. The average values obtained for the data accepted by these statistical tests are also presented in Tables 4, 5 and 6.

As can be seen from these tables, the ranges of reported values are extremely wide for some elements; the maximum to minimum value ratios approach 1000 for Mn and Sb and 2000 for Fe, Zn, As and Ag. Nevertheless, the degree of scatter of the data, indicated by percentage standard errors associated

with the overall averages is not excessively large for the majority of the elements listed. If an arbitrary value of 10% standard error is chosen as a measure for less scattered data, the measurements of the elements Mn, Fe, Cu, Zn, As, Se, Ag and Cd fall into the less-than-10% category. If we consider 50% standard error as a lower limit of the high degree of scatter, the measurements of Cr and Sb exceed this limit. The degree of scatter for the measurements of Co, Ni, Hg and Pb is considered as medium (standard errors between 10 and 50%). Similar trends are observed for the standard errors of the average values computed after rejecting outlying data by applying Chauvenet's or Dixon's test, although the scatter of the data was considerably reduced by such rejection. There was a tendency for Chauvenet's test to reject more values than Dixon's. In any case, these observations suggest that the average values obtained after rejecting outlying values for the 8 elements in the less-than-10%-standard-error category mentioned above can be regarded as the "consensus values" in the present intercalibration. It is reasonable to consider that these "consensus values" closely approach the actual concentrations of Mn, Fe, Cu, Zn, As, Se, Ag and Cd in the present sample. For the remaining 6 elements, Cr, Co, Ni, Sb, Hg and Pb, the corresponding average values should not be considered as the "consensus values" without reservation, due to the wider scatter of the data reported. Considering the difficulties involved in Pb analyses as well as the avoidance of contamination during analysis, any average value presented in Table 6 for Pb should not be regarded in any sense as representative.

3. Analytical methods used

In Tables 1, 2 and 3 only abbreviated descriptions of the analytical methods adopted by the participating laboratories are given. These show only the type of analytical methods used but do not indicate the variety of the procedures involved in each type of analytical method, i.e., atomic absorption spectrophotometry, neutron activation analysis, etc. A detailed survey of analytical methods with a view to correlating analytical procedures with the quality of the data obtained is in progress and the result of this survey will be available in the future. As a first step for correlating analytical methods used with the data reported, the results obtained by atomic absorption spectrometry (AAS) are compared with those obtained by neutron activation analysis (NAA) in Table 7 for 9 selected elements, Cr, Mn, Fe, Co, Cu, Zn, Ag, Cd and Hg. The number of reported results was insufficient to apply this comparison to other trace elements. Disregarding differences in detailed procedures existing among individual laboratories which used the same method, averages of the results obtained by each

technique were calculated after rejecting outlying data by applying Chauvenet's test. As can be seen in Table 7, the agreement between the averages obtained for AAS and NAA is good for 6 elements, Mn, Fe, Cu, Zn, Ag and Cd, all of which could be assigned "consensus values". This shows that both AAS and NAA give similarly reliable results for these elements at the concentration levels in the present sample. For the remaining elements, Cr, Co and Hg, it is reasonable to assume that the average values for NAA are closer to the actual concentrations, considering the fact that the AAS technique tends to produce systematically higher results for these elements.

The code number of your laboratory is ____.

TABLE 1 RESULTS OF Cr, Mn, Fe, Co AND Ni MEASUREMENTS ON OYSTER SAMPLE MA-M-1
BY DIFFERENT LABORATORIES ($\mu\text{g/g}$ -dry matter at 105-110 °C)

Code No.	Method	Cr	Mn	Fe	Co	Ni
1	NAA	-	-	311 $\pm$ 11	0.29 $\pm$ 0.011	-
2	NAA	-	-	270 $\pm$ 14	0.321 $\pm$ 0.003	<15
3	IDMS	-	-	-	-	-
4	AAS	-	-	-	N.D.	N.D.
5	FLM	-	-	-	-	-
5	NAA	-	0.094 $\pm$ (28%)	0.21 $\pm$ (40%)	-	-
6	NAA	-	75 $\pm$ 6	-	-	-
7	AAS-ESM	-	75.8	300	-	6.69
	ESM	2.06	-	-	<3	-
8	AAS	-	68 $\pm$ 3	220 $\pm$ 13	-	-
	SPM	-	-	-	-	-
9	ASV	-	-	-	-	-
10	AAS	4.36 $\pm$ (50%)	63.4 $\pm$ (20%)	244 $\pm$ (20%)	-	-
11	NAA	-	-	-	-	-
12	NAA	0.304 $\pm$ 0.05	-	281.8 $\pm$ 38.9	0.44 $\pm$ 0.068	-
13	NAA(chem)	-	68.2 $\pm$ 1.5	-	-	-
	NAA(inst)	0.86	-	294.1	0.43	-
14	AAS	N.D.	78 $\pm$ 1.60	360 $\pm$ 17.82	5.7 $\pm$ 0.77	6.9 $\pm$ 0.95
15	NAA	<2	58 $\pm$ 3	320 $\pm$ 30	0.37 $\pm$ 0.04	<20
16	XRF	-	42.9 $\pm$ (10%)	243 $\pm$ (10%)	-	-
17	AAS	2.0 $\pm$ (10%)	-	-	-	1.7 $\pm$ (10%)
	XRF	<5	80 $\pm$ (20%)	290 $\pm$ (20%)	<2	<5
	NAA	0.44 $\pm$ 0.24	$\geq$ 55	300 $\pm$ 80	0.33 $\pm$ 0.03	-
18	ASV	-	-	-	-	-
19	XESM	-	0.0207*	0.0870*	-	-
20	AAS	1.1 $\pm$ 0.2	58.5 $\pm$ 1.2	312 $\pm$ 12	1.45 $\pm$ 0.4	2.35 $\pm$ 0.4

* Relative value : Ratio to Zn concentration.

N.D. : Not detected or not determined due to too low concentration.

TABLE 1 (continued)

Code No.	Method	Cr	Mn	Fe	Co	Ni
21	AAS	-	74 $\pm(0.2\%)$	308 $\pm(0.9\%)$	-	-
22	NAA	-	-	-	-	-
23	AAS	-	-	-	-	-
24	AAS	1.6 $\pm(10\%)$	75 $\pm(10\%)$	438 $\pm(10\%)$	-	1.7 $\pm(10\%)$
	NAA	0.43 $\pm(25\%)$	75 $\pm(10\%)$	305 $\pm(15\%)$	0.39 $\pm(10\%)$	-
	ESM	-	110	510	-	-
25	NAA	-	-	-	-	-
26	AAS	-	-	-	-	-
27	XRF	-	45 ± 18	117 ± 12	-	-
28	NAA	-	71 $\pm(7\%)$	348 $\pm(5\%)$	0.32 $\pm(5\%)$	-
29	NAA	-	64 ± 3	315 ± 75	3.7 ± 0.5	-
30	NAA	-	70 ± 3	-	-	-
31	NAA	1.77 $\pm(20.5\%)$	-	-	0.53 $\pm(10\%)$	-
32	AAS	1.23 $\pm(10\%)$	67.2 $\pm(10\%)$	-	-	-
32	AAS	-	77 ± 1	308 ± 5	-	4.7 ± 0.3
33	XRF	-	71.3 ± 2.9	308 ± 12	-	-
	SEMS	-	-	-	Present	Present
34	NAA	0.842 ± 0.032	34.7 ± 3.6	317.3 ± 32.3	0.72 ± 0.044	4.17 ± 0.05
35	AAS	0.346 ± 0.027	-	312 ± 6.6	-	-
	NAA	-	-	453 ± 8	0.568 ± 0.013	-
36	AAS	2.52 $\pm(7.7\%)$	76.2 $\pm(0.55\%)$	254 $\pm(0.99\%)$	0.286 $\pm(17.73\%)$	3.41 $\pm(1.59\%)$
37	AAS	-	66.03	290.3	-	-
38	NAA	-	-	-	-	-
39	NAA	-	-	-	-	-
40	NAA	1.12 $\pm(6\%)$	74.77 $\pm(6\%)$	314.10 $\pm(6\%)$	0.346 $\pm(6\%)$	-
41	AAS	-	26.4 $\pm(10\%)$	-	-	1.18 $\pm(20\%)$
42	AAS	-	-	-	-	-
43	AAS	-	55.98 ± 4.45	-	4.26 ± 0.18	-
44	NAA(chem)	0.65 ± 0.36	-	310 ± 40	0.41 ± 0.04	<0.2
	NAA(inst)	≤ 6	81 ± 9	440 ± 50	0.56 ± 0.14	-
45	AAS	-	-	58	-	-

N.D. : Not detected or not determined due to too low concentration.

TABLE 1 (continued)

Code No.	Method	Cr	Mn	Fe	Co	Ni
46	AAS	-	-	-	-	-
47	AAS	-	-	-	-	-
48	NAA	0.55 $\pm$ (42.2%)	-	283.5 $\pm$ (3.79%)	0.37 $\pm$ (3.56%)	-
49	AAS	-	75.36	344.2	-	-
50	AAS	1.14 $\pm$ 0.09	76 $\pm$ 3	270 $\pm$ 20	0.38 $\pm$ 0.03	3.3 $\pm$ 0.4
	ASV	-	-	-	-	-
51	SSMS	7 $\pm$ (10~30%)	62 $\pm$ (10~30%)	390 $\pm$ (10~30%)	-	-
		75 $\pm$ (10~30%)	-	-	-	-
52	AAS	<10	70	390	-	22
53	AAS	1.57	-	-	-	-
54	AAS	1.0	-	-	-	-
55	AAS	-	-	-	-	-
56	AAS	-	-	35.4 $\pm$ (9.6%)	-	-
57	AAS	-	-	-	-	-
58	AAS	1.1 $\pm$ 0.41	-	-	-	4.9 $\pm$ 0.39
59	NAA	2.7 $\pm$ 1.3	-	475 $\pm$ 85	0.53 $\pm$ 0.09	N.D.
60	AAS	-	-	-	-	-

AAS : Atomic Absorption Spectrophotometry

ASV : Anodic Stripping Voltammetry

ESM : Emission Spectrometry

FLM : Fluorimetry

IDMS : Isotope Dilution Mass Spectrometry

NAA : Neutron Activation Analysis

NAA (chem) : NAA with radiochemical separation

NAA (inst) : Instrumental NAA

SPM : Spectrophotometry

SSMS : Spark Source Mass Spectrometry

XESM : X-Ray Emission Spectrometry

XRF : X-Ray Fluorescence Spectrometry

N.D. : Not detected or not determined due to too low concentration.

TABLE 2 RESULTS OF Cu, Zn, As, Se AND Ag MEASUREMENTS ON OYSTER SAMPLE MA-N-1
BY DIFFERENT LABORATORIES ($\mu\text{g/g}$ -dry matter at 105-110 °C)

Code No.	Method	Cu	Zn	As	Se	Ag
1	NAA	-	2900 $\pm$ 40	-	1.87 $\pm$ 0.049	+
2	NAA	-	2650 $\pm$ 30	11.4 $\pm$ 2.3	2.82 $\pm$ 0.18	6.55 $\pm$ 0.12
3	IDMS	-	-	-	-	-
4	AAS	333	2634	-	-	5.23
	FLM	-	-	-	1.90	-
5	NAA	-	2.8 $\pm$ (14%)	0.016 $\pm$ (49%)	-	0.0058 $\pm$ (31%)
6	NAA	336 $\pm$ 7	1734 $\pm$ 190	-	-	-
7	AAS-ESM	326	2630	9.58	0.075	7.82
	ESM	-	-	-	-	-
8	AAS	310 $\pm$ 10	2900 $\pm$ 200	-	-	-
	SPM	-	-	9.0 $\pm$ 0.3	-	-
9	ASV	-	261.6 $\pm$ (10%)	-	-	-
10	AAS	316 $\pm$ (15%)	2998 $\pm$ (15%)	-	-	2.43 $\pm$ (10%)
11	NAA	-	-	-	-	-
12	NAA	320 $\pm$ 16.45	2654 $\pm$ 169.8	-	0.88 $\pm$ 0.132	9.98 $\pm$ 1.28
13	NAA(chem)	331 $\pm$ 6	2950 $\pm$ 75	12.3 $\pm$ 0.3	2.26 $\pm$ 0.08	-
	NAA(inst)	-	2746	-	2.37	5.74
14	AAS	384 $\pm$ 14.60	3036 $\pm$ 122.36	-	-	6.8 $\pm$ 0.20
15	NAA	-	3000 $\pm$ 400	15 $\pm$ 2	2.0 $\pm$ 0.2	<5
16	XRF	243 $\pm$ (10%)	2278 $\pm$ (10%)	-	-	-
17	AAS	201 $\pm$ (2%)	2930 $\pm$ (2%)	-	-	6.0 $\pm$ (3%)
	XRF	350 $\pm$ (10%)	2860 $\pm$ (10%)	8 $\pm$ (20%)	-	-
	NAA	>300	3060 $\pm$ 300	-	2.6 $\pm$ 0.8	5.7 $\pm$ 1.0
18	ASV	350 $\pm$ (5%)	-	-	-	-
19	XESM	0.1086*	-	-	-	-
20	AAS	245 $\pm$ 3	2900 $\pm$ 30	-	-	-

* Relative value : Ratio to Zn concentration.

N.D. : Not detected or not determined due to too low concentration.

TABLE 2 (continued)

Code No.	Method	Cu		Zn		As		Se		Ag	
21	AAS	325	$\pm(4\%)$	2910	$\pm(2.7\%)$	-	-	-	-	-	-
22	NAA	-	-	-	-	-	-	-	-	-	-
23	AAS	354	± 14	3002	± 31	-	-	-	-	-	-
24	AAS	360	$\pm(5\%)$	2810	$\pm(10\%)$	-	-	-	5.6	$\pm(10\%)$	-
	NAA	313	$\pm(10\%)$	3020	$\pm(10\%)$	12	$\pm(10\%)$	-	7.0	$\pm(10\%)$	-
	ESM	430	-	-	-	-	-	-	7.7	-	-
25	NAA	-	-	44.59	-	26.23	-	2.79	-	-	-
26	AAS	326	$\pm(9\%)$	3060	$\pm(15\%)$	-	-	-	-	-	-
27	XRF	130	± 14	1535	± 153	-	-	-	-	-	-
28	NAA	-	-	2430	$\pm(3\%)$	-	-	2.4	$\pm(11\%)$	1.6	$\pm(20\%)$
29	NAA	-	-	2500	± 300	6.4	± 0.5	2.60	± 0.08	4.2	± 0.3
30	NAA	251	± 13	2730	± 98	11.6	± 0.21	2.36	± 0.25	-	-
31	NAA	-	-	2706.43	$\pm(10\%)$	11.34	$\pm(38\%)$	N.D.	-	5.68	$\pm(10\%)$
	AAS	307.91	$\pm(10\%)$	2286.4	$\pm(10\%)$	-	-	-	-	-	-
32	AAS	352	± 4	2970	± 30	-	-	-	-	-	-
33	XRF	410	± 14	2953	± 88	-	-	-	-	-	-
	SSMS	-	-	-	-	Present		-	-	~3	-
34	NAA	224.5	± 9.7	2617	± 37.5	-	-	0.49	± 0.056	7.27	± 0.33
35	AAS	333	± 4	3073	± 131	-	-	-	-	-	-
	NAA	-	-	4478	± 97	-	-	2.41	± 0.04	7.38	± 0.10
36	AAS	359	$\pm(0.55\%)$	2833	$\pm(0.35\%)$	-	-	-	-	4.46	$\pm(10.7\%)$
37	AAS	326.5	-	2705	-	-	-	-	-	-	-
38	NAA	254	$\pm(5.15\%)$	-	-	9.32	$\pm(5.3\%)$	-	-	-	-
39	NAA	-	-	-	-	-	-	-	-	-	-
40	NAA	356.4	$\pm(6\%)$	2772.2	$\pm(6\%)$	11.64	$\pm(6\%)$	1.89	$\pm(6\%)$	5.61	$\pm(6\%)$
41	AAS	319.0	$\pm(20\%)$	2861	$\pm(10\%)$	-	-	-	-	-	-
42	AAS	330	± 10	2500	± 100	-	-	-	-	-	-
43	AAS	-	-	-	-	-	-	-	-	-	-
44	NAA(chem)	400	± 80	2760	± 300	13	± 2	2.2	± 0.2	-	-
	NAA(inst)	-	-	3000	± 200	13	± 2	-	-	7.3	± 0.8
45	AAS	55.5	-	5400	-	-	-	-	-	-	-

N.D. : Not detected or not determined due to too low concentration.

TABLE 2 (continued)

Code No.	Method	Cu	Zn	As	Se	Ag
46	AAS	-	2686 $\pm$ (5.3%)	-	-	-
47	AAS	-	-	-	-	-
48	NAA	-	-	-	-	-
49	AAS	291.7	2989	-	-	5.4
50	AAS	340 $\pm$ 20	3240 $\pm$ 70	-	-	-
	ASV	269 $\pm$ 4	2790 $\pm$ 90	-	-	-
51	SSMS	295 $\pm$ (10~30%)	2150 $\pm$ (10~30%)	5 $\pm$ (10~30%)	$\leq$ 0.5	$\leq$ 1.2
52	AAS	290	1700	9.8	-	-
53	AAS	204 $\pm$ (14%)	3190 $\pm$ (14%)	-	-	-
54	AAS	335	2863	-	-	-
55	AAS	324.8 $\pm$ (1.9%)	2617 $\pm$ (1.2%)	-	-	-
56	AAS	381 $\pm$ (3.1%)	2703 $\pm$ (1.4%)	-	-	-
57	AAS	-	-	-	-	-
58	AAS	351 $\pm$ 2.6	3132 $\pm$ 21.53	-	-	-
59	NAA	190 $\pm$ 57	260 $\pm$ 4	0.93 $\pm$ 0.55	9.9 $\pm$ 0.35	4.7 $\pm$ 0.6
60	AAS	-	-	-	-	-

AAS : Atomic Absorption Spectrophotometry

ASV : Anodic Stripping Voltammetry

ESM : Emission Spectrometry

FLM : Fluorimetry

IDMS : Isotope Dilution Mass Spectrometry

NAA : Neutron Activation Analysis

NAA (chem) : NAA with radiochemical separation

NAA (inst) : Instrumental NAA

SPM : Spectrophotometry

SSMS : Spark Source Mass Spectrometry

XESM : X-Ray Emission Spectrometry

XRF : X-Ray Fluorescence Spectrometry

N.D. : Not detected or not determined due to too low concentration.

TABLE 3 RESULTS OF Cd, Sb, Hg AND Pb MEASUREMENTS ON OYSTER SAMPLE NA-M-1
BY DIFFERENT LABORATORIES ($\mu\text{g/g}$ -dry matter at 105-110 °C)

Code No.	Method	Cd	Sb	Hg	Pb
1	NAA	-	0.052 ± 0.009	0.26 ± 0.027	-
2	NAA	-	<0.021	<0.24	-
3	IDMS	-	-	-	$1.26 \sim 1.51$
4	AAS	2.33	-	-	N.D.
	FLM	-	-	-	-
5	NAA	-	-	-	-
6	NAA	-	-	-	-
7	AAS-ESM	1.86	-	0.108	0.94
	ESM	-	-	-	-
8	AAS	3.1 ± 0.8	-	0.16 ± 0.005	-
	SPM	-	-	-	-
9	ASV	-	-	-	$2.36 \pm (\sim 10\%)$
10	AAS	$1.87 \pm (25\%)$	-	-	$0.77 \pm (100\%)$
11	NAA	-	-	$0.82 \pm (10\%)$	-
12	NAA	2.40 ± 0.377	0.19 ± 0.08	-	-
13	NAA(chem)	-	-	-	-
	NAA(inst)	-	0.030	-	-
14	AAS	3.7 ± 0.18	-	-	12.7 ± 1.03
15	NAA	-	<0.9	-	-
16	XRF	-	-	-	-
17	AAS	$0.4 \pm (20\%)$	-	$0.30 \pm (2\%)$	$0.2 \pm (20\%)$
	XRF	-	-	<2	<3
	NAA	-	0.30 ± 0.08	1.6 ± 0.6	-
18	ASV	$2.26 \pm (1.4\%)$	-	-	$2.4 \pm (3.1\%)$
19	XESM	-	-	-	0.0117*
20	AAS	2.3 ± 0.12	-	0.61 ± 0.04	6.7 ± 0.7

* Relative value : Ratio to Zn concentration.

N.D. : Not detected or not determined due to low concentration.

TABLE 3 (continued)

Code No.	Method	Cd	Sb	Hg	Pb
21	AAS	-	-	0.169 $\pm$ (7.4%)	-
22	NAA	-	-	0.166 $\pm$ (6%)	-
23	AAS	3.36 $\pm$ 0.13	-	0.212 $\pm$ 0.014	1.67 $\pm$ 0.16
24	AAS	2.7 $\pm$ (10%)	-	0.16 $\pm$ (10%)	0.32 $\pm$ (20%)
	NAA	-	0.013	-	-
	ESM	-	-	-	-
25	NAA	-	-	-	-
26	AAS	3.3 $\pm$ (3%)	-	0.265 $\pm$ (9%)	2.1 $\pm$ (43%)
27	XRF	-	-	-	6 $\pm$ 5
28	NAA	-	-	-	-
29	NAA	-	-	-	-
30	NAA	2.33 $\pm$ 0.07	-	0.142 $\pm$ 0.007	-
31	NAA	-	N.D.	-	-
	AAS	-	-	0.135 $\pm$ (10%)	4.01 $\pm$ (10%)
32	AAS	-	-	-	-
33	XRF	-	-	-	-
	SSMS	~1	Present	-	~3
34	NAA	-	15.60 $\pm$ 1.5	-	-
35	AAS	-	-	-	2.74 $\pm$ 0.18
	NAA	-	-	-	-
36	AAS	2.12 $\pm$ (0.71%)	-	-	1.41 $\pm$ (7.6%)
37	AAS	-	-	-	-
38	NAA	1.82 $\pm$ (8.45%)	-	0.113 $\pm$ (9.45%)	-
39	NAA	2.45 $\pm$ (5%)	-	-	-
40	NAA	$\leq$ 4.68 $\pm$ (6%)	0.040 $\pm$ (7%)	0.154 $\pm$ (6%)	-
41	AAS	0.82 $\pm$ (10%)	-	-	4.7 $\pm$ (20%)
42	AAS	2.5 $\pm$ 0.1	-	0.21 $\pm$ 0.01	-
43	AAS	1.19 $\pm$ 0.03	-	-	-
44	NAA(chem)	1.9 $\pm$ 0.3	0.11 $\pm$ 0.04	0.18 $\pm$ 0.04	-
	NAA(inst)	-	-	-	-
45	AAS	-	-	-	-

N.D. : Not detected or not determined due to too low concentration.

TABLE 3 (continued)

Code No.	Method	Cd	Sb	Hg	Pb
46	AAS	-	-	-	-
47	AAS	1.94 $\pm$ 0.05	-	-	-
48	NAA	-	-	-	-
49	AAS	2.10	-	0.233	1.10
50	AAS	2.5 $\pm$ 0.2	-	0.154 $\pm$ 0.003	0.61 $\pm$ 0.04
	ASV	1.8 $\pm$ 0.1	-	0.170 $\pm$ 0.010	3.0 $\pm$ 0.2
51	SSMS	$\leq$ 0.15	$\leq$ 0.085	$\leq$ 0.07	$\leq$ 1.5
52	AAS	3.4	-	0.90	<20
53	AAS	2.1 $\pm$ (16%)	-	0.17 $\pm$ (4%)	-
54	AAS	2.67	-	0.16	1.22
55	AAS	<0.2	-	0.54 $\pm$ (<1%)	N.D.
56	AAS	4.4 $\pm$ (3.6%)	-	0.329 $\pm$ (6.1%)	7.6 $\pm$ (7.2%)
57	AAS	-	-	0.33 $\pm$ (4.54%)	-
58	AAS	3.3 $\pm$ 0.17	-	0.335 $\pm$ 0.027	5.7 $\pm$ 0.22
59	NAA	-	N.D.	-	-
60	AAS	-	-	0.218	-

N.D. : Not detected or not determined due to too low concentration.

AAS : Atomic Absorption Spectrophotometry

ASV : Anodic Stripping Voltammetry

ESM : Emission Spectrometry

FLM : Fluorimetry

IDMS : Isotope Dilution Mass Spectrometry

NAA : Neutron Activation Analysis

NAA (chem) : NAA with radiochemical separation

NAA (inst) : Instrumental NAA

SPM : Spectrophotometry

SSMS : Spark Source Mass Spectrometry

XESM : X-Ray Emission Spectrometry

XRF : X-Ray Fluorescence Spectrometry

TABLE 4 PRELIMINARY OVERALL AVERAGES AND AVERAGES AFTER CHAUVENET AND DIXON'S TESTS FOR THE RESULTS REPORTED FOR Cr, Mn, Fe, Co AND Ni MEASUREMENTS ON OYSTER SAMPLE MA-M-1

Element	Cr	Mn	Fe	Co	Ni
No. of Reported Results*	24	33	39	22	12
Range Reported ($\mu\text{g/g-dry}$)	0.304 - 75	0.094 - 110	0.21 - 510	0.29 - 5.7	1.18 - 22
Overall Average** ($\mu\text{g/g-dry}$)	5 ± 3 (60%)	66 ± 4 (6.1%)	300 ± 20 (6.7%)	1.0 ± 0.3 (30%)	5 ± 2 (40%)
<u>Chauvenet's Test</u>					
No. of Results accepted by the test	21	27	30	18	11
Average** ($\mu\text{g/g-dry}$)	1.2 ± 0.2 (17%)	71 ± 2 (2.8%)	304 ± 7 (2.3%)	0.43 ± 0.03 (6.9%)	3.7 ± 0.6 (1.7%)
<u>Dixon's Test</u>					
No. of Results accepted by the test	21	31	39	22	11
Average** ($\mu\text{g/g-dry}$)	1.2 ± 0.2 (17%)	67 ± 3 (4.5%)	300 ± 20 (6.7%)	1.0 ± 0.3 (30%)	3.7 ± 0.6 (18%)

* Results showing only detection limits of methods used are not included.

** The standard deviations given are 1 σ for mean (standard error).

TABLE 5 PRELIMINARY OVERALL AVERAGES AND AVERAGES AFTER CHAUVENET AND DIXON'S TESTS FOR THE RESULTS REPORTED FOR Cu, Zn, As, Se AND Ag MEASUREMENTS ON OYSTER SAMPLE MA-N-1

Element	Cu	Zn	As	Se	Ag
No. of Reported Results *	45	56	19	18	24
Range Reported ($\mu\text{g/g-dry}$)	55.5 - 430	2.8 - 5400	0.016 - 26.23	0.075 - 3.9	0.0058 - 9.98
Overall Average ** ($\mu\text{g/g-dry}$)	310 ± 10 (3.2%)	2630 ± 120 (4.6%)	10 ± 1 (10%)	2.1 ± 0.2 (9.5%)	5.6 ± 0.5 (8.9%)
<u>Chauvenet's Test</u>					
No. of Results accepted by the test	43	46	16	14	23
Average ** ($\mu\text{g/g-dry}$)	317 ± 8 (2.5%)	2820 ± 30 (1.1%)	10.5 ± 0.7 (6.7%)	2.32 ± 0.09 (3.9%)	5.8 ± 0.4 (6.9%)
<u>Dixon's Test</u>					
No. of Results accepted by the test	44	55	18	18	24
Average ** ($\mu\text{g/g-dry}$)	313 ± 9 (2.9%)	2600 ± 100 (3.8%)	9.4 ± 1.2 (11%)	2.1 ± 0.2 (9.5%)	5.6 ± 0.5 (8.9%)

* Results showing only detection limits of methods used are not included.

** The standard deviations given are 1 σ for mean (standard error).

TABLE 6 PRELIMINARY OVERALL AVERAGES AND AVERAGES AFTER CHAUVENET AND DIXON'S TESTS FOR THE RESULTS REPORTED FOR Cd, Sb, Hg and Pb MEASUREMENTS ON OYSTER SAMPLE MA-M-1

Element	Cd	Sb	Hg	Pb
No. of Reported Results**	30	8	29	23
Range Reported (µg/g-dry)	0.4-4.4	0.013-15.60	0.108-1.6	0.2-12.7
Overall Average*** (µg/g-dry)	2.3±0.2 (8.7%)	2±2 (100%)	0.32±0.06 (19%)	3.2±0.6 (19%)
<u>Chauvenet's Test</u>				
No. of Results accepted by the test	30	5	24	22
Average*** (µg/g-dry)	2.3±0.2 (8.7%)	0.05±0.02 (40%)	0.20±0.02 (10%)	2.7±0.5 (19%)
<u>Dixon's Test</u>				
No. of Results accepted by the test	30	7	28	22
Average*** (µg/g-dry)	2.3±0.2 (8.7%)	0.11±0.04 (36%)	0.28±0.04 (14%)	2.7±0.5 (19%)

** Results showing only detection limits of methods used are not included.

*** The standard deviations given are 1 σ for mean (standard error).

TABLE 7
COMPARISONS OF THE REPORTED RESULTS OBTAINED BY ATOMIC
ABSORPTION SPECTROPHOTOMETRY (AAS) WITH THOSE OBTAINED
BY NEUTRON ACTIVATION ANALYSIS (NAA)

Element	Method	No. of Results Reported	No. of Results in Chauvenet's Range	Average in Chauvenet's Range ($\mu\text{g/g-dry}$)
Cr	AAS	11	10	1.4 $\pm$ 0.2
	NAA	10	8	0.7 $\pm$ 0.1
Mn	AAS	16	15	70 $\pm$ 2
	NAA	11	9	71 $\pm$ 2
Fe	AAS	16	13	300 $\pm$ 10
	NAA	17	12	302 $\pm$ 5
Co	AAS	5	5	2.4 $\pm$ 1.1
	NAA	17	16	0.44 $\pm$ 0.03
Cu	AAS	27	23	334 $\pm$ 5
	NAA	10	10	300 $\pm$ 20
Zn	AAS	28	25	2890 $\pm$ 40
	NAA	21	21	2500 $\pm$ 300
Ag	AAS	8	7	5.9 $\pm$ 0.4
	NAA	14	11	6.1 $\pm$ 0.3
Cd	AAS	22	22	2.5 $\pm$ 0.2
	NAA	5	5	2.2 $\pm$ 0.1
Hg	AAS	19	16	0.21 $\pm$ 0.02
	NAA	8	5	0.15 $\pm$ 0.01

ANNEX

LIST OF PARTICIPATING LABORATORIES

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PROGRESS REPORT No. 15
INTERCALIBRATION OF ANALYTICAL METHODS
ON MARINE ENVIRONMENTAL SAMPLES

International Laboratory of Marine Radioactivity, IAF
Principality of Monaco
June 1977

Preliminary Report on the Measurements of Trace Elements in Sea Plant and Copepod Samples.

In 1976 preparations of the sea plant sample (SP-M-1) and the copepod sample (MA-A-1) for trace element measurements were completed. The homogeneity of these samples was found satisfactory. Using these samples the intercalibration exercises of trace element measurements were organized with support of UNEP.

The sea plant sample (SP-M-1) was distributed to 98 laboratories and the copepod sample (MA-A-1) to 89 laboratories. Some laboratories took part in the measurements of one or the other of the samples, while 80 laboratories received both samples at the same time in the period of Dec. 1976 - May 1977. By June 1977, 55 laboratories had already reported the results of their measurements. Since requests to participate in the present intercalibration exercise are still coming in, the present exercise is expected to continue for some time before its completion. This means that the report will be excessively delayed if we await the recovery of the results of the whole group. Since a sufficient number of the results have already been sent in, it is considered appropriate to prepare a preliminary report on the basis of the results already at hand, especially for those who have already submitted results. In this way the participants will be able to judge the success of this intercalibration exercise, even though the compilation in the report is incomplete. A list of the 55 laboratories which submitted the results by 15 June 1977 is attached as the Annex.

1. Results reported

Although results from 55 laboratories were received by 15 June 1977, the compilation includes the data of only 52 laboratories. Despite the fact that the measurements of many elements were made, only the results reported on 14 selected trace elements, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Ag, Cd, Sb, Hg and Pb have been compiled at present. Tables 1, 2 and 3 contain the results on the sea plant sample (SP-M-1) and Tables 4, 5 and 6 those on the copepod sample (MA-A-1). In the compilation the following procedures were adopted:

- (a) All values given in the tables were taken from the original reports of the participants with as little change as possible; significant figures of the values given in the tables are identical to those of the data reported by the participants.
- (b) In cases where laboratories had not given laboratory averages for their individual measurements, arithmetical averages of the individual data were calculated and presented in the tables.
- (c) The associated errors of the measurements were reported either in absolute values or in percentages. The percent errors are given in parentheses in the tables.
- (d) In cases where laboratory averages were calculated at Monaco, associated errors for these averages were computed on the basis of the errors given for individual data when two or three data had been given for one element; where four or more data were available for calculating the average, standard errors were computed from the individual data reported.
- (e) In each table abbreviated descriptions of the analytical methods used are also given. These show only the type of analytical methods adopted, but do not indicate the variety of the procedures involved in each type of analytical method. The abbreviations used are indicated at the end of Table 6.

2. Preliminary summary of the data

In Table 7 the preliminary overall averages of the results reported from 52 laboratories on the above-mentioned 14 selected trace elements in the SP-M-1 and MA-A-1 samples are given. It should be emphasized that the table was intended to show the scatter of reported results in the present intercalibration exercises, but not to indicate "probable concentrations" of the trace elements in question in the samples. Since statistical criteria to reject outlying data were not applied, the overall averages do not, in any sense, represent the probable concentrations. It was observed, however, that the scatter of some elements was less than that of the others. For example, standard errors of the overall averages of several elements are less than 10% while those of all others exceed 10%, some of which exceed 30%.

- the elements with less than 10% standard error:
 - for SP-M-1 : Cr, Mn, Fe, Ni, Cu, Zn, Se, Sb, Pb;
 - for MA-A-1 : Fe, As, Se;
- the elements with more than 30% standard error:
 - for SP-M-1 : Ag, Cd, Hg;
 - for MA-A-1 : Cr, Co, Cu, Zn, Ag, Sb, Hg, Pb.

These observations indicate that the performances of the analyses for specific elements are apparently quite dependent on the matrices of the samples. Even for the elements with less than 10% standard error, the probable concentrations which will be estimated later may be different by a factor of as much as 2 for the overall averages presented in Table 7.

The code number of your laboratory is _____.

TABLE 1 RESULTS OF TRACE ELEMENT MEASUREMENTS ON THE SEA PLANT SAMPLE SP-M-1
(Cr, Mn, Fe, Co, Ni $\mu\text{g/g}$ dry matter)

Code No.	Method	Cr	Mn	Fe	Co	Ni
1	AAS	-	-	-	-	-
2	AAS	-	54.5 $\pm$ 0.7	1870 $\pm$ 20	1.91 $\pm$ 0.20	-
3	AAS	-	57.0 $\pm$ 1.9	1570 $\pm$ 27	-	17.2 $\pm$ 0.62
4	FT	-	-	-	-	-
5	AAS	5.96 $\pm$ 0.18	52.36 $\pm$ 0.91	-	-	16.45 $\pm$ 0.75
6	NAA	2.9 $\pm$ (10%)	43.5 $\pm$ (7%)	1360 $\pm$ (15%)	2.2 $\pm$ (10%)	-
7	AAS	1.4 $\pm$ (6%)	57.0 $\pm$ (8%)	-	0.186 $\pm$ (15%)	19.1 $\pm$ (5%)
8	Not Partic.	-	-	-	-	-
9	NAA	-	63.4 $\pm$ 1.6	1890 $\pm$ 33.1	-	-
10	NAA	5.07 $\pm$ 0.55	69 $\pm$ 6	1760 $\pm$ 90	2.10 $\pm$ 0.11	17.6 $\pm$ 1.8
	XRF	7.9 $\pm$ 2.8	69.7 $\pm$ 3.2	2220 $\pm$ 30	-	23.6 $\pm$ 0.8
11	NAA	3.34 $\pm$ 0.22	-	1661 $\pm$ 91	1.54 $\pm$ 0.07	-
12	AAS	11.7	41.1	1003.7	7.2	24.2
13	AAS	5.3 $\pm$ (10%)	-	1630 $\pm$ (5%)	-	-
14	NAA	-	55.6 $\pm$ 2.9	1967 $\pm$ 53	3.16 $\pm$ 0.22	-
15	AAS	<5	54.9	1860	-	23.8
16	AAS	2.41 $\pm$ (7%) 2.52 $\pm$ (5%)	64 $\pm$ (8%)	1950 $\pm$ (4%)	-	-
17	Not Partic.	-	-	-	-	-
18	AAS	-	-	-	-	-
19	AAS	4.0 $\pm$ 0.55	50.2 $\pm$ 3.4	1967 $\pm$ 20.0	5.87 $\pm$ 0.89	-
20	NAA	4.3 $\pm$ 0.44	53.9 $\pm$ 3.24	2180 $\pm$ 131	3.7 $\pm$ 0.3	-
21	NAA	4.56 $\pm$ 1.98	(AAS)	1622 $\pm$ (1.61%)	1.98 $\pm$ (1.3%)	-
22	Not Partic.	-	-	-	-	-
23	AAS	-	56 $\pm$ (5%)	-	-	-
	ESM	1 $\pm$ (30%)	-	-	1 $\pm$ (30%)	12 $\pm$ (30%)
	SPM	-	-	1560 $\pm$ (2.5%)	-	-
24	AAS	-	73.24 $\pm$ (1.0%)	-	-	-
25	AAS	4.2 $\pm$ (8%)	100 $\pm$ (6%)	1750 $\pm$ (3.7%)	<2	12 $\pm$ (27%)
26	NAA(inst)	4.2 $\pm$ 0.7	58.5 $\pm$ 2.1	2080 $\pm$ 64	2.04 $\pm$ 0.05	-
	NAA(chem)	-	64.1 $\pm$ 1.53	-	-	-
27	AAS	-	44.2 $\pm$ 0.2	1450 $\pm$ 50	-	20.4 $\pm$ 0.2
28	NAA	1.9 $\pm$ (15%)	-	1500 $\pm$ (15%)	1.1 $\pm$ (15%)	-
29	AAS	4.65 $\pm$ (12%)	63.66 $\pm$ (4%)	-	-	-
30	AAS	-	-	-	-	-

TABLE 1 (continued)

Code No.	Method	Cr	Mn	Fe	Co	Ni
31	AAS	--	40.3 $\pm(8.1\%)$	1357.4 $\pm(26.2\%)$	4.0 $\pm(16.1\%)$	18.1 $\pm(9.6\%)$
32	AAS	--	--	--	--	--
33	NAA	--	--	2230 $\pm(3\%)$	--	17.1 $\pm(18\%)$
34	AAS	2.23 $\pm(9.22\%)$	63.1 $\pm(0.74\%)$	1189 $\pm(1.06\%)$	1.09 $\pm(8.85\%)$	25.0 $\pm(1.26\%)$
35		--	59.25	1680	6.54	41.4
36	Not Partic.	--	--	--	--	--
37	AAS	--	--	--	--	--
38	NAA	--	--	1503 $\pm(3.73\%)$	--	--
39	AAS	--	60.3 $\pm(4.8\%)$	1250 $\pm(5.0\%)$	--	--
40	XESM	--	--	1740 ± 150	--	--
41	XRF	--	88 ± 6	3334 ± 238	--	--
42	NAA	4.7 $\pm(15\%)$	--	1900 $\pm(11\%)$	2.1 $\pm(11\%)$	--
43	NAA	5 $\pm(20\%)$	62 $\pm(10\%)$	1875 $\pm(10\%)$	2.5 $\pm(10\%)$	--
	AAS	5.1 $\pm(5-10\%)$	65 $\pm(2\%)$	2050 $\pm(2\%)$	2.2 $\pm(5\%)$	16.3 $\pm(5-10\%)$
44	ESM	4.3 $\pm(19\%)$	96.8 $\pm(9.6\%)$	--	<4.0	18.2 $\pm(14.2\%)$
45	AAS	3.5 $\pm(10\%)$	53 $\pm(5\%)$	2030 $\pm(5\%)$	= --	14.9 $\pm(10\%)$
46	AAS	--	--	--	--	--
47	AAS	--	--	--	--	--
48	AAS	4.80 ± 0.4	52 ± 2	1735 ± 35	3.77 ± 0.02	--
49	ASV	--	--	--	--	--
50	AAS	4.9 ± 0.2	71.3 ± 0.9	2070 ± 30	--	27.2 ± 0.5
51	AAS	--	--	--	--	--
52	NAA	--	--	--	--	--

TABLE 2 RESULTS OF TRACE ELEMENT MEASUREMENTS ON THE SEA PLANT SAMPLE SP-1
(Cu, Zn, As, Se, Ag $\mu\text{g/g}$ dry matter)

Code No.	Method	Cu	Zn	As	Se	Ag
1	AAS	-	-	-	-	-
2	AAS	11.8 $\pm$ 0.4	60.3 $\pm$ 0.5	-	-	-
3	AAS	11.2 $\pm$ 0.25	53.7 $\pm$ 1.38	-	-	-
4	FT	-	-	-	-	-
5	AAS	11.10 $\pm$ 0.21	104.30 $\pm$ 19	-	-	-
6	NAA	-	35 $\pm$ (15%)	1.83 $\pm$ (10%)	-	-
7	AAS	7.7 $\pm$ (6%)	63.1 $\pm$ (3%)	-	-	-
8	Not Partic.	-	-	-	-	-
9	NAA	11.2 $\pm$ 0.3	52.1 $\pm$ 2.3	3.06 $\pm$ 0.06	0.21 $\pm$ 0.01	-
10	NAA	<400	61.3 $\pm$ 4.2	3.6 $\pm$ 1.2	0.181 $\pm$ 0.055	0.46 $\pm$ 0.05
	XRF	15.9 $\pm$ 1.1	69.2 $\pm$ 1.3	5.09 $\pm$ 0.31	<0.56	-
11	NAA	-	55.3 $\pm$ 2.6	-	0.17 $\pm$ 0.03	-
12	AAS	11.2	48.4	-	-	-
13	AAS	10.3 $\pm$ (5%)	67.2 $\pm$ (5%)	-	-	-
14	NAA	15.7 $\pm$ 1.2	15.6 $\pm$ 1.6	-	-	-
15	AAS	16.4	58	-	-	-
16	AAS	15.5 $\pm$ (6%)	60 $\pm$ (6%)	-	-	-
17	Not Partic.	-	-	-	-	-
18	AAS	11.0	4.6	3.5	-	-
19	AAS	13.7 $\pm$ 1.0	63.4 $\pm$ 4.3	-	-	0.79 $\pm$ 0.24
20	NAA	-	55.34 $\pm$ 2.77	-	0.20 $\pm$ 0.017	2.7 $\pm$ 0.021
	AAS	8.56 $\pm$ 0.77	-	-	-	-
21	NAA	-	58.7 $\pm$ (1.60%)	-	0.170 $\pm$ (29.8%)	-
22	Not Partic.	-	-	-	-	-
23	AAS	11 $\pm$ (5%)	61 $\pm$ (5%)	-	-	-
	ESM	-	-	-	-	1 $\pm$ (30%)
	SPM	-	-	-	-	-
24	AAS	12.62 $\pm$ (4.6%)	58.21 $\pm$ (1.0%)	-	-	-
25	AAS	43.0 $\pm$ (12%)	225 $\pm$ (2.9%)	-	-	8.0 $\pm$ (23%)
26	NAA(inst)	-	90 $\pm$ 4	6.8 $\pm$ 1.1	0.14 $\pm$ 0.01	-
	NAA(chem)	11.0 $\pm$ 0.738	-	5.87 $\pm$ 0.537	-	-
27	AAS	12.0 $\pm$ 0.2	56 $\pm$ 2.0	-	-	1.3 $\pm$ 0.2
28	NAA	12 $\pm$ (15%)	48 $\pm$ (15%)	2.9 $\pm$ (15%)	0.19 $\pm$ (15%)	-
29	AAS	13.10 $\pm$ (14%)	76.88 $\pm$ (14%)	3.71 $\pm$ (9%)	-	-
30	AAS	8.5 $\pm$ 0.3	58 $\pm$ 3	-	-	-

TABLE 2 (continued)

Code No.	Method	Cu	Zn	As	Se	Ag
31	AAS	12.4	60.2 $\pm$ (1.7%)	-	-	-
32	AAS	9.3 $\pm$ (15%)	56.9 $\pm$ (4%)	-	-	-
33	NAA	-	77.2 $\pm$ (4%)	-	-	-
34	AAS	12.5 $\pm$ (1.09%)	65.6 $\pm$ (1.76%)	-	-	1.78 $\pm$ (2.84%)
35		14.61	95.21	-	-	-
36	Not Partic.	-	-	-	-	-
37	AAS	-	-	-	-	-
38	NAA	9.76 $\pm$ (5.33%)	52.6 $\pm$ (2.73%)	-	-	-
39	AAS	-	67.3 $\pm$ (2.2%)	-	-	-
40	XESM	-	-	-	-	-
41	XRF	23.3 $\pm$ 4.9	96 $\pm$ 12	-	-	-
42	NAA	-	59 $\pm$ (11%)	-	-	-
43	NAA	12 $\pm$ (10%)	61 $\pm$ (10%)	3.8 $\pm$ (10%)	-	0.60 $\pm$ (10%)
	AAS	16.9 $\pm$ (5%)	67 $\pm$ (5%)	-	-	-
44	ESM	13.2 $\pm$ (7.6%)	91.6 $\pm$ (10.7%)	-	-	<4.0
45	AAS	11.8 $\pm$ (5%)	68 $\pm$ (5%)	-	-	0.54 $\pm$ (15%)
46	AAS	-	-	-	-	-
47	AAS	9.0 $\pm$ (10%)	57.7 $\pm$ (10%)	-	-	-
48	AAS	11.4 $\pm$ 0.6	91 $\pm$ 5	11 $\pm$ 1	-	1.05 $\pm$ 0.1
49	ASV	8.1 $\pm$ (6%)	-	-	-	-
50	AAS	14.8 $\pm$ 0.5	73 $\pm$ 1	-	-	-
51	AAS	12 $\pm$ 0.5	52 $\pm$ 2	-	-	-
52	NAA	-	44.93 $\pm$ 2.98	2.38 $\pm$ 0.30	-	-

TABLE 3 RESULTS OF TRACE ELEMENT MEASUREMENTS ON THE SEA PLANT SAMPLE SP-M-1
(Cd, Sb, Hg, Pb $\mu\text{g/g}$ dry matter)

Code No.	Method	Cd	Sb	Hg	Pb
1	AAS	-	-	0.20 $\pm$ 0.03	-
2	AAS	0.44 $\pm$ 0.03	-	-	16.1 $\pm$ 0.6
3	AAS	0.36 $\pm$ 0.006	-	-	15.8 $\pm$ 0.26
4	FT	-	-	-	-
5	AAS	0.18 $\pm$ 0.05	-	-	-
6	NAA	-	-	-	-
7	AAS	0.39 $\pm$ (7%)	-	-	10.5 $\pm$ (6%)
8	Not Partic.	-	-	-	-
9	NAA	0.39 $\pm$ 0.01	-	0.31 $\pm$ 0.01	-
10	NAA	<3.7	0.40 $\pm$ 0.01	0.446 $\pm$ 0.021	-
	XRF	-	-	-	19.4 $\pm$ 0.8
11	NAA	-	0.527 $\pm$ 0.071	0.36 $\pm$ 0.05	-
12	AAS	1.7	0.50 $\pm$ 0.07	-	25.5
13	AAS	-	-	-	10.6 $\pm$ (15%)
14	NAA	-	-	-	-
15	AAS	0.43	-	-	14
16	AAS	0.505 $\pm$ (1%)	-	0.375 $\pm$ (4%)	23.9 $\pm$ (12%)
17	Not Partic.	-	-	-	-
18	AAS	0.56	-	0.07	16.8
19	AAS	0.88 $\pm$ 0.08	-	0.275 $\pm$ 0.005	18.3 $\pm$ 1.8
20	NAA	3.84 $\pm$ 0.384	0.80 $\pm$ 0.087	-	-
	AAS	-	-	-	-
21	NAA	-	0.620 $\pm$ (5.03%)	0.331 $\pm$ (7.3%)	-
22	Not Partic.	-	-	-	-
23	AAS	0.113 $\pm$ (10%)	-	0.250 $\pm$ (8%)	2.000 $\pm$ (10%)
	ESM	-	-	-	-
	SFM	-	-	-	-
24	AAS	0.43 $\pm$ (4.6%)	-	0.43 $\pm$ (0.0%)	14.04 $\pm$ (3.0%)
25	AAS	24 $\pm$ (14%)	-	<0.02	19 $\pm$ (13%)
26	NAA(inst)	-	0.87 $\pm$ 0.18	0.56 $\pm$ 0.03	-
	NAA(chem)	-	0.95 $\pm$ 0.050	0.55	-
27	AAS	1.3 $\pm$ 0.1	-	-	23.4 $\pm$ 1.0
28	NAA	0.50 $\pm$ (15%)	0.49 $\pm$ (15%)	5.6 $\pm$ (15%)	-
29	AAS	0.22 $\pm$ (8%)	-	0.34 $\pm$ (4%)	6.65 $\pm$ (18%)
30	AAS	0.5 $\pm$ 0.1	-	0.38 $\pm$ 0.01	14 $\pm$ 2

TABLE 3 (continued)

Code No.	Method	Cd	Sb	Hg	Pb
31	AAS	-	-	-	18.9 $\pm$ (12.9%)
32	AAS	0.222 $\pm$ (14%)	-	0.351 $\pm$ (8%)	20.4 $\pm$ (15%)
33	NAA	-	0.714 $\pm$ (7%)	-	-
34	AAS	0.95 $\pm$ (3.48%)	-	-	14.8 $\pm$ (1.65%)
35		-	-	-	-
36	Not Partic.	-	-	-	-
37	AAS	-	-	0.48 $\pm$ 0.03	-
38	NAA	0.28 $\pm$ (9.11%)	-	0.449 $\pm$ (7.49%)	-
39	AAS	-	-	0.331 $\pm$ (1.5%)	-
40	XESM	-	-	-	-
41	XRF	-	-	-	-
42	NAA	-	0.64 $\pm$ (12%)	-	-
43	NAA	-	0.6 $\pm$ (15%)	-	-
	AAS	1.11 $\pm$ (5~10%)	-	0.295 $\pm$ (5~10%)	27.0 $\pm$ (5~10%)
44	ESM	-	<4.0	-	13.6 $\pm$ (12%)
45	AAS	0.30 $\pm$ (25%)	-	0.30 $\pm$ (10%)	3.5 $\pm$ (10%)
46	AAS	-	-	-	-
47	AAS	0.56 $\pm$ (10%)	-	0.16 $\pm$ (10%)	5.8 $\pm$ (10%)
48	AAS	2.80 $\pm$ 0.3	-	0.200 $\pm$ 0.02	13.8 $\pm$ 1.4
49	ASV	0.14 $\pm$ (17%)	-	-	10.1 $\pm$ (14%)
50	AAS	-	-	-	27.0 $\pm$ 0.6
51	AAS	<0.2	-	0.24 $\pm$ 0.01	<1
52	NAA	-	-	0.159 $\pm$ 0.013	-

TABLE 4 RESULTS OF TRACE ELEMENT MEASUREMENTS ON THE COPEPOD SAMPLE MA-A-1
(Cr, Mn, Fe, Co, Ni $\mu\text{g/g}$ -dry matter)

Code No.	Method	Cr	Mn	Fe	Co	Ni
1	AAS	-	-	-	-	-
2	AAS	-	4.63 ± 0.38	73.9 ± 2.7	-	-
3	Not Partic.	-	-	-	-	-
4	FT	-	-	-	-	-
5	AAS	0.92 ± 0.09	2.61 ± 0.17	-	-	1.37 ± 0.16
6	NAA	-	5.6	$74 \pm (20\%)$	-	-
7	Not Partic.	-	-	-	-	-
8	AAS	0.82	-	-	-	1.49
9	NAA	-	2.68 ± 0.02	-	-	-
10	NAA	0.34 ± 0.07	<30	48.9 ± 3.7	0.099 ± 0.009	1.26 ± 0.27
	XRF	<3.2	3.7 ± 1.0	54.7 ± 3.1	-	1.5 ± 0.5
11	NAA	-	-	56.4 ± 7.1	0.086 ± 0.008	-
12	AAS	5.4	2.9	53.8	1.5	4.3
13	Not Partic.	-	-	-	-	-
14	NAA	-	0.83 ± 0.09	134 ± 3.5	-	-
15	AAS	<5	2.8	56	-	2.0
16	Not Partic.	-	-	-	-	-
17	AAS	-	-	66.8 ± 8.8	-	-
18	AAS	-	-	-	-	-
19	Not Partic.	-	-	-	-	-
20	NAA	0.24 ± 0.036	-	64.7 ± 4.2	0.175 ± 0.014	-
	AAS	-	-	-	-	-
21	NAA	$1.54 \pm (12.2\%)$	-	$82.7 \pm (8.67\%)$	$0.150 \pm (7.1\%)$	-
22	AAS	-	2.7 ± 0.2	59.9 ± 5.0	-	3.0 ± 0.5
23	Not Partic.	-	-	-	-	-
24	AAS	-	$3.22 \pm (6.2\%)$	-	-	-
25	Not Partic.	-	-	-	-	-
26	Not Partic.	-	-	-	-	-
27	AAS	-	2.6 ± 0.2	55 ± 5	-	2.2 ± 0.2
28	NAA	$0.083 \pm (25\%)$	-	$56 \pm (15\%)$	$0.041 \pm (15\%)$	-
29	AAS	$1.10 \pm (12\%)$	$3.57 \pm (4\%)$	-	-	-
30	AAS	-	-	-	-	-

TABLE 4 (continued)

Code No.	Method	Cr	Mn	Fe	Co	Ni
31	AAS	-	2.3 $\pm$ (18.2%)	51.3 $\pm$ (13.4%)	N.D.	1.8 $\pm$ (29.3%)
32	AAS	-	-	-	-	-
33	NAA	-	-	95.1 $\pm$ (21%)	0.147 $\pm$ (5%)	-
34	AAS	0.380 $\pm$ (34.4%)	3.20 $\pm$ (2.94%)	90.2 $\pm$ (3.05%)	0.098 $\pm$ (14.00%)	3.00 $\pm$ (8.33%)
35	Not Partic.	-	-	-	-	-
36	AAS	1.42 $\pm$ 0	1.42 $\pm$ 0	49.13 $\pm$ 1.58	-	-
37	AAS	-	-	-	-	-
38	NAA	-	-	51.2 $\pm$ (6.20%)	-	-
39	AAS	-	<10	44 $\pm$ (8.4%)	-	-
40	XESM	-	-	170 $\pm$ 90	-	-
41	XRF	-	2.2 $\pm$ 0.7	50.1 $\pm$ 4.6	-	-
42	NAA	-	-	-	-	-
43	NAA	-	-	-	-	-
	AAS	-	-	-	-	-
44	Not Partic.	-	-	-	-	-
45	AAS	1	1.9 $\pm$ (20%)	82 $\pm$ (15%)	-	1.2 $\pm$ (15%)
46	AAS	-	9.3 $\pm$ 3.2	-	N.D.	-
47	AAS	-	-	-	-	-
48	AAS	6.90 $\pm$ 0.5	2.2 $\pm$ 0.2	54 $\pm$ 1	0.63 $\pm$ 0.02	-
49	Not Partic.	-	-	-	-	-
50	AAS	-	4.5 $\pm$ 0.6	55 $\pm$ 2	-	-
51	AAS	-	-	-	-	-
52	NAA	-	-	-	-	-

TABLE 5 RESULTS OF TRACE ELEMENT MEASUREMENTS ON THE COPEPOD SAMPLE MA-A-1
(Cu, Zn, As, Se, Ag $\mu\text{g/g}$ dry matter)

Code No.	Method	Cu	Zn	As	Se	Ag
1	AAS	-	-	-	-	-
2	AAS	9.52 $\pm$ 0.08	185 $\pm$ 1	-	-	-
3	Not Partic.	-	-	-	-	-
4	FT	-	-	-	-	-
5	AAS	8.43 $\pm$ 0.65	246.77 $\pm$ 25	-	-	-
6	NAA	-	103	4.63	-	-
7	Not Partic.	-	-	-	-	-
8	AAS	7.54	146	7.9	-	0.34
9	NAA	6.77 $\pm$ 0.12	144 $\pm$ 7	8.67 $\pm$ 0.40	3.00 $\pm$ 0.14	-
10	NAA	<300	145 $\pm$ 12	10.2 $\pm$ 0.5	3.21 $\pm$ 0.15	0.085 $\pm$ 0.012
	XRF	7.6 $\pm$ 0.6	158 $\pm$ 3	8.17 $\pm$ 0.23	2.05 $\pm$ 0.37	-
11	NAA	-	156.4 $\pm$ 3.1	-	3.03 $\pm$ 0.07	-
12	AAS	9.4	152.1	-	-	-
13	Not Partic.	-	-	-	-	-
14	NAA	8.47 $\pm$ 0.69	162 $\pm$ 7.5	-	-	-
15	AAS	10.5	160	-	-	-
16	Not Partic.	-	-	-	-	-
17	AAS	10.4	155 $\pm$ 3	-	-	-
18	AAS	7.3	13.4	7.0	-	-
19	Not Partic.	-	-	-	-	-
20	NAA	-	162.4 $\pm$ 5.1	-	4.4 $\pm$ 0.37	0.582 $\pm$ 0.0425
	AAS	-	-	-	-	-
21	NAA	-	176.5 $\pm$ (0.45%)	7.69 $\pm$ (19.18%)	3.39 $\pm$ (5.0%)	0.090 $\pm$ (23.6%)
22	AAS	7.52 $\pm$ 1.0	160.5 $\pm$ 10	-	-	-
23	Not Partic.	-	-	-	-	-
24	AAS	7.73 $\pm$ (2.6%)	154.52 $\pm$ (3.7%)	-	-	-
25	Not Partic.	-	-	-	-	-
26	Not Partic.	-	-	-	-	-
27	AAS	7.7 $\pm$ 0.1	159 $\pm$ 2.0	-	-	0.9 $\pm$ 0.2
28	NAA	6.4 $\pm$ (15%)	128 $\pm$ (15%)	7.1 $\pm$ (15%)	3.3 $\pm$ (15%)	-
29	AAS	8.33 $\pm$ (14%)	186.8 $\pm$ (14%)	2.73 $\pm$ (9%)	-	-
30	AAS	4.9 $\pm$ 0.2	171 $\pm$ 2	-	-	-

TABLE 5 (continued)

Code No.	Method	Cu	Zn	As	Se	Ag
31	AAS	7.4 $\pm$ (11.1%)	151.2 $\pm$ (5.9%)	-	-	-
32	AAS	8.64 $\pm$ (21%)	134.28 $\pm$ (4%)	-	-	-
33	NAA	-	184 $\pm$ (4%)	-	-	-
34	AAS	8.48 $\pm$ (1.85%)	185 $\pm$ (1.87%)	-	-	0.516 $\pm$ (8.47%)
35	Not Partic.	-	-	-	-	-
36	AAS	7.86 $\pm$ 0.31	172.7 $\pm$ 0	-	-	-
37	AAS	-	-	-	-	-
38	NAA	6.63 $\pm$ (2.58%)	141 $\pm$ (1.46%)	-	-	-
39	AAS	-	163 $\pm$ (1.3%)	-	-	-
40	XESM	130 $\pm$ 50	2600 $\pm$ 200	-	-	-
41	XRF	7.1 $\pm$ 1.1	169 $\pm$ 3	-	-	-
42	NAA	-	-	-	-	-
43	NAA	-	-	-	-	-
	AAS	-	-	-	-	-
44	Not Partic.	-	-	-	-	-
45	AAS	6.8 $\pm$ (25%)	169 $\pm$ (3%)	-	-	0.07 $\pm$ (30%)
46	AAS	9.27 $\pm$ 1.2	136.9 $\pm$ 0.5	-	-	1.48 $\pm$ 0.6
47	AAS	7.9 $\pm$ (10%)	159.4 $\pm$ (10%)	-	-	-
48	AAS	6.0 $\pm$ 0.3	175 $\pm$ 10	8.5 $\pm$ 0.8	-	0.26 $\pm$ 0.03
49	Not Partic.	-	-	-	-	-
50	AAS	9.5 $\pm$ 0.4	165 $\pm$ 2	-	-	-
51	AAS	7.0 $\pm$ 0.5	140 $\pm$ 5	-	-	-
52	NAA	-	130.5 $\pm$ 7.5	11.02	2.55 $\pm$ 0.06	-

TABLE 6 RESULTS OF TRACE ELEMENT MEASUREMENTS ON THE COPEPOD SAMPLE MA-A-1
(Cd, Sb, Hg, Pb $\mu\text{g/g}$ -dry matter)

Code No.	Method	Cd	Sb	Hg	Pb
1	AAS	-	-	<0.05	-
2	AAS	0.678 $\pm$ 0.012	-	-	1.10 $\pm$ 0.14
3	Not Partic.	-	-	-	-
4	FT	-	-	-	-
5	AAS	0.5 $\pm$ 0.03	-	-	-
6	NAA	-	-	-	-
7	Not Partic.	-	-	-	-
8	AAS	0.84	-	<0.04	2.8
9	NAA	0.57 $\pm$ 0.02	0.014 $\pm$ 0.001	0.237 $\pm$ 0.007	-
10	NAA	<4.2	0.012 $\pm$ 0.003	0.219 $\pm$ 0.047	-
	XRF	-	-	-	<1.8
11	NAA	-	0.19 $\pm$ 0.06	0.29 $\pm$ 0.07	-
12	AAS	1.1	-	-	4.9
13	Not Partic.	-	-	-	-
14	NAA	-	-	-	-
15	AAS	0.70	-	-	1.1
16	Not Partic.	-	-	-	-
17	AAS	0.60 $\pm$ 0.01	-	-	-
18	AAS	0.77	-	0.21	<1.0
19	Not Partic.	-	-	-	-
20	NAA	3.01 $\pm$ 0.301	0.0347 $\pm$ 0.0038	-	-
	AAS	-	-	-	-
21	NAA	-	0.188 $\pm$ (15.9%)	0.357 $\pm$ (14.5%)	-
22	AAS	1.15 $\pm$ 0.3	-	-	1.22 $\pm$ 0.7
23	Not Partic.	-	-	-	-
24	AAS	0.63 $\pm$ (27%)	-	0.30 $\pm$ (3.3%)	1.29 $\pm$ (12.4%)
25	Not Partic.	-	-	-	-
26	Not Partic.	-	-	-	-
27	AAS	1.1 $\pm$ 0.1	-	-	<1.0
28	NAA	0.61 $\pm$ (15%)	0.016 $\pm$ (25%)	3.6 $\pm$ (15%)	-
29	AAS	0.67 $\pm$ (8%)	-	0.27 $\pm$ (4%)	0.87 $\pm$ (18%)
30	AAS	0.73 $\pm$ 0.04	-	0.28 $\pm$ 0.01	2.9 $\pm$ 0.3

TABLE 6 (continued)

Code No.	Method	Cd	Sb	Hg	Pb
31	AAS	-	-	-	N.D.
32	AAS	0.618 $\pm$ (13%)	-	0.253 $\pm$ (10%)	2.0 $\pm$ (5%)
33	NAA	-	-	-	-
34	AAS	0.925 $\pm$ (5.04%)	-	-	1.04 $\pm$ (11.35%)
35	Not Partic.	-	-	-	-
36	AAS	0.55 $\pm$ 0	-	-	0.62 $\pm$ 0.13
37	AAS	-	-	0.32 $\pm$ 0.03	-
38	NAA	0.78 $\pm$ (13.6%)	-	0.358 $\pm$ (6.70%)	-
39	AAS	-	-	0.264 $\pm$ (4.9%)	-
40	XESM	-	-	-	-
41	XRF	-	-	-	-
42	NAA	-	-	-	-
43	NAA	-	-	-	-
	AAS	-	-	-	-
44	Not Partic.	-	-	-	-
45	AAS	0.54 $\pm$ (20%)	-	0.10 $\pm$ (20%)	-
46	AAS	N.D.	-	-	11.4 $\pm$ 1.1
47	AAS	1.1 $\pm$ (10%)	-	0.15 $\pm$ (10%)	1.1 $\pm$ (10%)
48	AAS	1.28 $\pm$ 0.07	-	$\leq$ 0.05	1.4 $\pm$ 0.4
49	Not Partic.	-	-	-	-
50	AAS	0.68 $\pm$ 0.08	-	0.31 $\pm$ 0.02	-
51	AAS	< 0.2	-	0.25 $\pm$ 0.01	< 1
52	NAA	-	-	0.164 $\pm$ 0.022	-

AAS : Atomic Absorption Spectrophotometry

ASV : Anodic Stripping Voltammetry

ESM : Emission Spectrometry

NAA : Neutron Activation Analysis

NAA (chem) : NAA with radiochemical separation

NAA (inst) : NAA Instrumental

FT : Fission Track

SPM : Spectrophotometry

XESM : X-Ray Emission Spectrometry

XRF : X-Ray Fluorescence Spectrometry

IP : Inverse Polarography

N.D. : Not detected or not determined
due to too low concentration

Not Partic. : Not Participated

TABLE 7 PRELIMINARY OVERALL AVERAGE VALUES FOR THE RESULTS OF TRACE
ELEMENT MEASUREMENTS REPORTED ON SP-M-1 AND MA-A-1 SAMPLES
(15 June 1977)

Element	No. of values reported	SP-M-1		MA-A-1		
		Overall average ^{**} ($\mu\text{g/g-dry}$)		No. of values reported	Overall average ^{**} ($\mu\text{g/g-dry}$)	
Cr	26	4.3	± 0.4 (9.3%)	11	1.7	± 0.7 (41%)
Mn	32	61	± 3 (4.9%)	20	3.2	± 0.4 (13%)
Fe	34	1790	± 70 (3.9%)	25	69	± 6 (8.7%)
Co	20	2.8	± 0.4 (14%)	9	0.3	± 0.2 (67%)
Ni	18	20	± 2 (10%)	11	2.1	± 0.3 (14%)
Cu	36	13	± 1 (7.7%)	28	12	± 4 (33%)
Zn	44	66	± 5 (7.6%)	37	220	± 70 (32%)
As	12	4.5	± 0.7 (16%)	11	7.6	± 0.7 (9.2%)
Se	7	0.18	± 0.01 (5.6%)	8	3.1	± 0.2 (5.5%)
Ag	10	1.8	± 0.7 (39%)	9	0.5	± 0.2 (40%)
Cd	26	1.7	± 0.9 (53%)	23	0.9	± 0.1 (11%)
Sb	11	0.65	± 0.05 (7.7%)	6	0.08	± 0.04 (50%)
Hg	27	0.5	± 0.2 (40%)	18	0.4	± 0.2 (50%)
Pb	26	16	± 1 (6.3%)	14	2.4	± 0.8 (33%)

^{**}The errors given are those in the standard deviations of the means
(standard errors).

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PROGRESS REPORT No. 16
INTERCALIBRATION OF ANALYTICAL METHODS
ON MARINE ENVIRONMENTAL SAMPLES

International Laboratory of Marine Radioactivity
I.A.E.A.
Principality of Monaco
March 1978

Trace Element Measurements on Oyster Homogenate (MA-M-1)
(Supplement)

The results of the preliminary survey, made on the data reported before June 1976 for trace elements in the oyster sample (MA-M-1), have already been presented in Progress Report No. 13 (PR-13). Since that time further results have been received from a number of laboratories. Altogether 125 laboratories received the oyster sample, of which 87 actually reported the results of their measurements to date. In the present report, the results are given of a survey on the reported data, including those received after the publication of the previous survey.

Only individual results reported from 27 laboratories on the 14 selected trace elements (as selected in PR-13) are given here as supplements to Table 1 (for Cr, Mn, Fe, Co and Ni), Table 2 (for Cu, Zn, As, Se and Ag) and Table 3 (for Cd, Sb, Hg and Pb) in PR-13, since those reported earlier from 60 laboratories were already presented. A list of these 27 laboratories with the names of those who were involved in the analytical work is given as an Annex. The abbreviations for the type of analytical techniques used by the laboratories, given in Tables 1, 2 and 3 (supplement), are as follows:

AAS: Atomic Absorption Spectrophotometry
ASV: Anodic Stripping Voltammetry
DPP: Differential Pulse Polarography
ESM: Emission Spectrometry
FLM: Fluorometry
NAA: Neutron Activation Analysis
PAA: Proton Activation Analysis
XES: X-Ray Emission Spectrometry
XRF: X-Ray Fluorescence Spectrometry

The procedures adopted for compiling individual data are similar to those described in PR-13.

Tables 4, 5 and 6 show ranges and overall averages for the concentration of the 14 selected trace elements in the oyster sample, based on all the data reported by the 87 laboratories. In these tables average values, obtained after rejecting outlying data by applying Chauvenet's or Dixon's test, are also presented. These tables should replace the corresponding tables given in PR-13, since they are now based on the complete set of data available in the present inter-calibration exercise.

The inclusion of the additional data in the survey has brought about, however, only minor changes to the general conclusions drawn in PR-13 on the tendencies of the data scatter for each element, the relationship between the "consensus value" and true concentration, etc. Nevertheless, the average concentrations for Cr, Mn, Fe, Co, Cu, Zn, Se, Ag, Cd, Hg and Pb, computed after applying Chauvenet's test, are now considered to approach closely the true concentrations of these elements in the oyster sample. The consideration is based on comparisons of these average concentrations with the "probable concentrations" estimated from the data reported by selected laboratories. For Ni, As and Sb the situation is not clear from the data available at present.

The survey presented here has been possible through a joint effort on the part of all participating laboratories. The endeavour of all those who contributed to the present intercalibration exercise is gratefully acknowledged.

TABLE 1 RESULTS OF Cr, Mn, Fe, Co AND Ni MEASUREMENTS ON OYSTER SAMPLE MA-M-1
BY DIFFERENT LABORATORIES ($\mu\text{g/g}$ -dry matter at 105-110 °C)
(SUPPLEMENT)

Code No.	Method	Cr	Mn	Fe	Co	Ni
61	AAS	-	85 $\pm(5\%)$	2800 $\pm(5\%)$	-	-
	ESM	<2.5	-	-	<0.7	<0.7
62	NAA	-	73 ± 2.4	-	-	-
	AAS	-	73 $\pm(2\sim3\%)$	316 $\pm(2\sim3\%)$	-	-
	PAA	<10	75 $\pm(10\%)$	320 $\pm(30\%)$	-	<3.0
63	AAS	-	79	353	-	-
64	AAS	1.1 ± 0.03	40.0	242 ± 1	-	-
65	AAS	-	-	-	-	-
	FLM	-	-	-	-	-
66	NAA	-	-	-	-	-
67	AAS	<0.5	79 $\pm(3\%)$	279 $\pm(11\%)$	<0.5	<0.5
68	AAS	-	67.3 $\pm(4\%)$	-	-	-
69	DPP	-	-	-	-	-
	AAS	-	-	-	-	-
70	ESM	2.4	-	-	0.7	2.4
71	AAS	<5	63.3 $\pm(5\%)$	240 $\pm(5\%)$	-	0.27 $\pm(10\%)$
72	AAS	-	-	-	-	-
73	AAS	3.7 $\pm(10\%)$	72 $\pm(3\%)$	323 $\pm(3\%)$	-	2.9 $\pm(10\%)$
74	NAA	-	-	1002 ± 200	0.45 ± 0.11	-
75	AAS	-	-	-	-	-
76	ASV	-	-	-	-	-
77	AAS	-	-	-	0.78 $\pm(10\%)$	-
78	AAS	-	-	-	-	-
79	AAS	3.63 ± 0.18	70.2 ± 0.6	312 ± 11	-	5.48
80	AAS	-	76.8 ± 1	223.7 ± 5	<4.67	-
81	XRF	-	67 ± 7.6	284 ± 8	-	-
82	AAS	1 ± 0.1	77 ± 2	327 ± 5	0.490 ± 0.02	-
83	AAS	-	79 $\pm(5\%)$	-	-	-
84	AAS	0.56 $\pm(30\%)$	81 $\pm(5\%)$	346 $\pm(3\%)$	<0.02	0.83 $\pm(3\%)$
85	XES	-	-	170 ± 90	-	-
86	AAS	-	-	-	-	-
87	AAS	-	-	-	-	-

TABLE 2 RESULTS OF Cu, Zn, As, Se AND Ag MEASUREMENTS ON OYSTER SAMPLE MA-M-1
BY DIFFERENT LABORATORIES ($\mu\text{g/g}$ dry matter at 105-110 °C)
(SUPPLEMENT)

Code No.	Method	Cu		Zn		As	Se	Ag
61	AAS	175	$\pm(5\%)$	140	$\pm(5\%)$	-	-	-
	ESM	-	-	-	-	-	-	7 $\pm(20\%)$
62	NAA	-	-	-	-	-	-	-
	AAS	345	$\pm(2-3\%)$	2680	$\pm(2-3\%)$	-	-	-
	PAA	-	-	3415	$\pm(10\%)$	13 $\pm(10\%)$	-	-
63	AAS	367	-	3080	-	-	-	-
64	AAS	303.0	± 0.1	2400	-	-	-	-
65	AAS	-	-	-	-	-	-	-
	FLM	-	-	-	-	-	2.1	-
66	NAA	280	$\pm(15\%)$	2500	$\pm(15\%)$	14 $\pm(15\%)$	2.5 $\pm(15\%)$	-
67	AAS	325	$\pm(2\%)$	2849	$\pm(4\%)$	-	-	-
68	AAS	307	$\pm(4.1\%)$	2885.5	$\pm(2.4\%)$	-	-	-
69	DPP	304	-	-	-	-	-	-
	AAS	-	-	-	-	-	-	-
70	ESM	480	-	-	-	-	-	-
71	AAS	325	$\pm(5\%)$	2791	$\pm(5\%)$	-	-	-
72	AAS	-	-	-	-	-	-	-
73	AAS	331	$\pm(3\%)$	3032	$\pm(3\%)$	-	-	6.1 $\pm(10\%)$
74	NAA	-	-	3285	± 394	163 ± 21	0.05 ± 0.01	-
75	AAS	-	-	-	-	-	-	-
76	ASV	280	$\pm(10\%)$	3020	$\pm(10\%)$	-	-	-
77	AAS	386.3	$\pm(5\%)$	3186	$\pm(10\%)$	-	-	-
78	AAS	-	-	-	-	-	-	-
79	AAS	328	± 14	2737	± 40	-	-	-
80	AAS	337.3	± 1	2595.3	± 59	-	-	17.66 ± 2.63
81	XRF	309	± 23	2820	± 26	-	-	-
82	AAS	306	± 5	2842	± 20	-	-	5.40 ± 0.3
83	AAS	351	$\pm(5\%)$	3629	$\pm(6\%)$	-	-	-
84	AAS	340	$\pm(5\%)$	4120	$\pm(2\%)$	7.5 $\pm(3\%)$	-	-
85	XES	130	± 50	2600	± 200	-	-	-
86	AAS	365	$\pm(13.32\%)$	2759	$\pm(11.47\%)$	-	-	-
87	AAS	-	-	-	-	-	-	-

TABLE 3 RESULTS OF Cd, Sb, Hg AND Pb MEASUREMENTS ON OYSTER SAMPLE MA-M-1
BY DIFFERENT LABORATORIES ($\mu\text{g/g}$ dry matter at 105-110 °C)
(SUPPLEMENT)

Code No.	Method	Cd	Sb	Hg	Pb
61	AAS	-	-	-	-
	ESM	-	-	-	0.7 $\pm$ (20%)
62	NAA	2.85 $\pm$ 0.10	-	-	-
	AAS	-	-	-	-
	PAA	<5	<5	-	-
63	AAS	2.5	-	-	-
64	AAS	2.2 $\pm$ 0.02	-	0.16 $\pm$ 0.01	1.18
65	AAS	-	-	0.23	-
	FLM	-	-	-	-
66	NAA	2.4 $\pm$ (15%)	-	0.18 $\pm$ (15%)	-
67	AAS	1.37 $\pm$ (10%)	-	0.14 $\pm$ (7%)	1.59 $\pm$ (25%)
68	AAS	2.5 $\pm$ (5.88%)	-	0.200 $\pm$ (9.15%)	-
69	DPP	1.93	-	-	1.31
	AAS	1.90	-	0.251	-
70	ESM	-	-	-	-
71	AAS	0.66 $\pm$ (5%)	-	-	1.34 $\pm$ (10%)
72	AAS	1.40 $\pm$ (10%)	-	0.205 $\pm$ (12%)	-
73	AAS	3.0 $\pm$ (10%)	-	-	<0.5
74	NAA	-	-	-	-
75	AAS	-	-	0.059	-
76	ASV	2.3 $\pm$ (10%)	-	-	1.2 $\pm$ (10%)
77	AAS	2.16 $\pm$ (10%)	-	0.09 $\pm$ (10%)	1.1 $\pm$ (10%)
78	AAS	-	-	0.1967	-
79	AAS	2.13 $\pm$ 0.14	-	0.26 $\pm$ 0.01	2.26 $\pm$ 0.21
80	AAS	2.51 $\pm$ 0.13	-	-	12.73 $\pm$ 0.8
81	XRF	-	-	-	-
82	AAS	21 $\pm$ 2	-	0.160 $\pm$ 0.03	2.13 $\pm$ 0.2
83	AAS	2.5 $\pm$ (6%)	-	0.131 $\pm$ (6%)	-
84	AAS	2.135 $\pm$ (3%)	-	<0.01	-
85	XES	-	-	-	-
86	AAS	3.458 $\pm$ (15.79%)	-	0.192 $\pm$ (9.55%)	-
87	AAS	1.92 $\pm$ 0.03	-	0.171 $\pm$ 0.02	2.19 $\pm$ 0.15

TABLE 4 OVERALL AVERAGES AND AVERAGES AFTER CHAUVENET AND DIXON'S TESTS
FOR THE RESULTS REPORTED FOR Cr, Mn, Fe, Co AND Ni MEASUREMENTS
ON OYSTER SAMPLE MA-M-1

Element	Cr	Mn	Fe	Co	Ni
No. of Reported Results*	30	49	54	26	17
Range reported ($\mu\text{g/g-dry}$)	0.304 - 75	0.094 - 110	0.21 - 2800	0.29 - 5.7	0.27 - 22
Overall Average ** ($\mu\text{g/g-dry}$)	4 ± 2 (50%)	68 ± 2 (2.9%)	360 ± 50 (14%)	1.0 ± 0.3 (30%)	4 ± 1 (25%)
<u>Chauvenet's Test</u>					
No. of Results accepted by the test	25	42	42	22	16
Average ** ($\mu\text{g/g-dry}$)	1.2 ± 0.1 (8.3%)	72 ± 1 (1.4%)	302 ± 6 (2.0%)	0.46 ± 0.03 (6.5%)	3.3 ± 0.5 (15%)
<u>Dixon's Test</u>					
No. of Results accepted by the test	28	47	52	26	16
Average ** ($\mu\text{g/g-dry}$)	1.5 ± 0.2 (13%)	69 ± 2 (2.9%)	300 ± 10 (3.3%)	1.0 ± 0.3 (30%)	3.3 ± 0.5 (15%)

* Results showing only detection limits of methods used are not included.

** The standard deviations given are 1 σ for mean (standard error).

TABLE 5 OVERALL AVERAGES AND AVERAGES AFTER CHAUVENET AND DIXON'S TESTS
FOR THE RESULTS REPORTED FOR Cu, Zn, As, Se AND Ag MEASUREMENTS
ON OYSTER SAMPLE MA-K-1

Element	Cu	Zn	As	Se	Ag
No. of Reported Results *	66	77	23	21	28
Range Reported ($\mu\text{g/g-dry}$)	55.5 - 480	2.8 - 5400	0.016 - 163	0.05 - 3.9	0.0058 - 17.66
Overall Average ** ($\mu\text{g/g-dry}$)	311 ± 9 (2.9%)	2700 ± 100 (3.7%)	17 ± 7 (41%)	2.0 ± 0.2 (10%)	6.1 ± 0.6 (9.8%)
<u>Chauvenet's test</u>					
No. of Results accepted by the test	60	64	19	21	26
Average ** ($\mu\text{g/g-dry}$)	322 ± 6 (1.9%)	2830 ± 30 (1.1%)	10.7 ± 0.6 (5.6%)	2.0 ± 0.2 (10%)	5.8 ± 0.4 (6.9%)
<u>Dixon's Test</u>					
No. of Results accepted by the test	66	77	21	21	27
Average ** ($\mu\text{g/g-dry}$)	311 ± 9 (2.9%)	2700 ± 100 (3.7%)	10 ± 1 (10%)	2.0 ± 0.2 (10%)	5.6 ± 0.4 (7.1%)

* Results showing only detection limits of methods used are not included.

** The standard deviations given are 1 σ for mean (standard error).

TABLE 6 OVERALL AVERAGES AND AVERAGES AFTER CHAUVENET AND DIXON'S TESTS
FOR THE RESULTS REPORTED FOR Cd, Sb, Hg AND Pb MEASUREMENTS ON
OYSTER SAMPLE MA-M-1

Element	Cd	Sb	Hg	Pb
No. of Reported Results*	50	8	44	34
Range Reported ($\mu\text{g/g-dry}$)	0.4 - 21	0.013 - 15.60	0.059 - 1.6	0.2 - 12.73
Overall Average** ($\mu\text{g/g-dry}$)	2.7 ± 0.4 (15%)	2 ± 2 (100%)	0.27 ± 0.04 (15%)	3.0 ± 0.5 (17%)
<u>Chauvenet's Test</u>				
No. of Results accepted by the test	48	5	39	26
Average** ($\mu\text{g/g-dry}$)	2.2 ± 0.1 (4.5%)	0.05 ± 0.02 (40%)	0.19 ± 0.01 (5.3%)	1.5 ± 0.2 (13%)
<u>Dixon's Test</u>				
No. of Results accepted by the test	49	7	39	32
Average** ($\mu\text{g/g-dry}$)	2.3 ± 0.1 (4.3%)	0.11 ± 0.04 (36%)	0.19 ± 0.01 (5.3%)	2.3 ± 0.3 (13%)

* Results showing only detection limits of methods used are not included.

** The standard deviations given are 1 σ for mean (standard error).

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PROGRESS REPORT No. 16
INTERCALIBRATION OF ANALYTICAL METHODS
ON MARINE ENVIRONMENTAL SAMPLES

International Laboratory of Marine Radioactivity, IAEA
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October 1978

Preliminary Report on the Measurements of Trace Elements in Fish Flesh

During 1977 the fish flesh homogenate sample (MA-A-2) was prepared for trace element measurements. The homogenization of this sample was much more difficult than that for the other samples, oyster, copepod etc. The fish sample (MA-A-2) was distributed to 106 laboratories between April - June 1978. By the end of September 1978 results were received from 56 laboratories. A preliminary compilation of the results reported is given in the present report. The final compilation and survey of the results will be made when more results are received. A list of the 56 laboratories which submitted results up to September 1978 is attached as an Annex.

1. Results reported

Although results from 56 laboratories were received by September 1978, the compilation includes the data from only 54 laboratories. Results of measurements of more than 40 elements were reported, but the present compilation has been made for only 14 selected elements, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Ag, Cd, Sb, Hg and Pb. Tables 1, 2 and 3 contain respectively the compilation for [Cr, Mn, Fe, Co, Ni], [Cu, Zn, As, Se, Ag] and [Cd, Sb, Hg, Pb]. As has been done in the past, the following procedures were adopted:

- (a) All values given in the tables were taken from the original reports of the participants with as little change as possible; significant figures of the values given in the tables are identical to those of the data reported by the participants.
- (b) In cases where laboratories had not given laboratory averages for their individual measurements, arithmetical averages of the individual data were calculated and presented in the tables.

- (c) The associated errors of the measurements were reported either in absolute values or in percentages. The percent errors are given in parentheses in the tables.
- (d) In cases where laboratory averages were calculated at Monaco, associated errors for these averages were computed on the basis of the errors given for individual data when two or three data had been given for one element; where four or more data were available for calculating the average, standard errors were computed from the individual data reported.
- (e) In each table abbreviated descriptions of the analytical methods used are also given. These show only the type of analytical methods adopted, but do not indicate the variety of the procedures involved in each type of analytical method. The abbreviations used are as follows:

AAS: Atomic Absorption Spectrophotometry
ASV: Anodic Stripping Voltammetry
NAA: Neutron Activation Analysis
SSMS: Spark Source Mass Spectrometry
XESM: X-ray Emission Spectrometry
XRF: X-ray Fluorescence Spectrometry

2. Preliminary summary of the data

Table 4 gives the preliminary overall averages of the results reported on the 14 selected trace elements from 54 laboratories. This table is intended to show the relative scatter of the reported results in the present intercalibration exercise, but does not indicate "probable concentrations" of trace elements in this sample. As statistical tests to reject outliers were not applied, the overall averages presented in Table 4 do not, in any case, represent the "probable concentrations". It was observed, however, that the average values, based on less scattered data for some elements, for example, those associated with less than $\pm 10\%$ standard error, may be close to the "probable concentrations" (e.g. Mn, Fe, Cu, Zn, As and Hg). On the other hand, the average values associated with large standard errors, which exceed $\pm 20\%$, may be several times off the probable concentrations of the elements (e.g. Cr, Co, Ag, Cd, Sb, Pb).

The code number of your laboratory is: _____

TABLE 1 RESULT OF TRACE ELEMENT MEASUREMENTS ON THE FISH FLESH SAMPLE MA-A-2
(Cr, Mn, Fe, Co, Ni $\mu\text{g/g}$ -dry matter)

Code No.	Method	Cr	Mn	Fe	Co	Ni
1	AAS	-	< 0.08	-	-	-
2	AAS	-	-	-	-	-
3	ASV	-	-	-	-	-
3	AAS	1.4 $\pm$ (10%)	-	-	-	< 1.1 $\pm$ (10%)
4	AAS	-	1 $\pm$ 0.5	70 $\pm$ 10	-	-
5	AAS	-	-	-	-	-
6	AAS	0.54 $\pm$ 0.08	N.D.	98 $\pm$ 14	-	-
7	XESM	-	-	68 $\pm$ (50%)	-	-
8	AAS	-	-	-	-	-
9	AAS	-	-	-	-	0.70 $\pm$ (10%)
10	AAS	-	-	-	-	-
11	AAS	-	-	-	-	0.51 $\pm$ 0.020
12	AAS	-	1.3 $\pm$ 0.64	-	-	-
13	AAS	4.7 $\pm$ 0.8	0.6 $\pm$ 0.1	50 $\pm$ 2	0.03 $\pm$ 0.01	0.5 $\pm$ 0.1
14	AAS	-	0.44 $\pm$ 0.05	50 $\pm$ 2	-	-
	ASV	-	-	-	-	-
15	AAS	-	-	-	-	-
16	AAS	-	1.1 $\pm$ 0.06	-	-	-
	NAA	2.1 $\pm$ 0.16	0.7 $\pm$ 0.05	67.7 $\pm$ 4.5	0.070 $\pm$ 0.002	-
17	NAA	2.08 $\pm$ (9.5%)	-	76.5 $\pm$ (7.7%)	0.135 $\pm$ (9.9%)	-
18	AAS	-	0.960 $\pm$ (11.29%)	-	-	-
19	AAS	-	-	-	-	-
20	XRF	2.0 $\pm$ 0.5	-	50.0 $\pm$ 5	-	1.0 $\pm$ 0.3
21	AAS	-	0.75 $\pm$ (5%)	53.3 $\pm$ (5%)	-	< 1.5
22	AAS	-	-	49.2 $\pm$ 3.5	-	-
23	AAS	0.87 $\pm$ (14%)	0.7 $\pm$ (36%)	56.3 $\pm$ (7%)	1.7 $\pm$ (26%)	2.5 $\pm$ (52%)
24	AAS	4.07 $\pm$ 0.44	-	50.0 $\pm$ 12.3	-	1.48 $\pm$ 0.16
25	XESM	12.9 $\pm$ 1.2	3.5 $\pm$ 0.9	62.5 $\pm$ 1.5	-	2.5 $\pm$ 0.4
26	AAS	1.40 $\pm$ (10.0%)	0.91 $\pm$ (5.5%)	-	-	-
27	AAS	-	-	50.9 $\pm$ 1.4	-	-
28	AAS	-	N.D	-	-	-
29	AAS	< 0.3	< 1.0	35.6	-	0.81
30	XRF	-	-	82 $\pm$ 15	-	-

TABLE 1 (continued)

Code No.	Method	Cr	Mn	Fe	Co	Ni
31	NAA	-	0.587 $\pm$ 0.010	53.7 $\pm$ 2.2	-	-
32	AAS	0.950 $\pm$ 0.12	1.180 $\pm$ 0.25	158.15 $\pm$ 18.11	-	-
33	AAS	-	-	-	-	-
34	AAS	1.04 $\pm$ (4%)	0.89 $\pm$ (2%)	59.3 $\pm$ (9%)	0.097 $\pm$ (9%)	1.00 $\pm$ (5%)
35	NAA	3.06 $\pm$ (3.5%)	0.85 $\pm$ (4%)	59.7 $\pm$ (5%)	0.11 $\pm$ (6%)	-
36	AAS	5.34 $\pm$ (10%)	1.78 $\pm$ (5%)	49.1 $\pm$ (5%)	2.19 $\pm$ (5%)	-
37	AAS	-	0.645 $\pm$ (10%)	-	-	-
38	AAS	0.49 $\pm$ (13%)	0.63 $\pm$ (6%)	58 $\pm$ (7%)	-	0.42 $\pm$ (9%)
39	NAA	1.31 $\pm$ (12.1%)	0.75 $\pm$ (8.9%)	61 $\pm$ (10.1%)	0.061 $\pm$ (16.9%)	-
40	AAS	-	-	47.0 $\pm$ (8.4%)	0.147 $\pm$ (26.9%)	0.137 $\pm$ (8.9%)
41	NAA	-	-	-	-	-
42	AAS	1.1 $\pm$ (10%)	1.0 $\pm$ (20%)	50.9 $\pm$ (10%)	0.6 $\pm$ (10%)	0.7 $\pm$ (10%)
43	AAS	1.6 $\pm$ (20%)	0.8 $\pm$ (20%)	-	0.09 $\pm$ (20%)	2.5 $\pm$ (20%)
	NAA	1.4 $\pm$ (20%)	0.51 $\pm$ (10%)	67 $\pm$ (20%)	0.054 $\pm$ (25%)	-
44	AAS	-	-	-	-	-
45	AAS	-	0.81	65	-	1.20
46	AAS	-	-	56.7 $\pm$ (4%)	-	-
	NAA	5.71 $\pm$ 0.13	0.72 $\pm$ 0.04	57.5 $\pm$ 5.4	0.057 $\pm$ 0.006	$\leq$ 1.7
47	NAA	9.9 $\pm$ (1.5%)	-	67.3 $\pm$ (3.8%)	0.064 $\pm$ (5.4%)	-
48	NAA	-	-	-	-	-
49	AAS	-	-	58.9 $\pm$ 0.7	-	-
	NAA	-	-	-	0.082 $\pm$ 0.020	-
50	AAS	-	-	-	-	-
51	AAS	2 $\pm$ 0.3	0.9 $\pm$ 0.2	47 $\pm$ 5	$\leq$ 0.1	0.9 $\pm$ 0.1
	NAA	2.0 $\pm$ 0.2	0.5 $\pm$ 0.05	34 $\pm$ 4	-	-
	SSMS	1.4 $\pm$ (30%)	0.5 $\pm$ (30%)	-	-	0.8 $\pm$ (30%)
52	AAS	3.59 $\pm$ 1.20	2.00 $\pm$ 0.80	56.61 $\pm$ 8.12	1.65 $\pm$ 0.55	2.54 $\pm$ 0.59
53	AAS	-	-	-	-	-
54	AAS	-	-	43 $\pm$ 9	0.18 $\pm$ 0.01	0.72 $\pm$ 0.05
55	NAA	1.22 $\pm$ 0.21	$<$ 0.7	47.9 $\pm$ 1.6	0.044 $\pm$ 0.001	$<$ 1
	XRF	1.4 $\pm$ 0.6	1.1 $\pm$ 0.4	51.8 $\pm$ 3.4	$<$ 0.8	0.89 $\pm$ 0.16
56	AAS	0.74 $\pm$ 0.08	0.64 $\pm$ 0.05	48.7 $\pm$ 2.0	0.03 $\pm$ 0.015	0.63 $\pm$ 0.10

TABLE 2
RESULT OF TRACE ELEMENT MEASUREMENTS ON THE FISH FLESH SAMPLE MA-A-2
(Cu, Zn, As, Se, Ag $\mu\text{g/g}$ -dry matter)

Code No.	Method	Cu	Zn	As	Se	Ag
1	AAS	3.49 $\pm$ (9.7%)	32.32 $\pm$ (5.3%)	-	1.23 $\pm$ (3.2%)	-
2	AAS	4.2 $\pm$ 0.4	33.3 $\pm$ 1.0	-	-	-
	ASV	-	-	-	-	-
3	AAS	3.9 $\pm$ (10%)	33 $\pm$ (10%)	< 0.1 $\pm$ (20%)	0.78 $\pm$ (20%)	-
4	AAS	3.5 $\pm$ 1	25 $\pm$ 1	-	-	-
5	AAS	-	-	2.74 $\pm$ (10%)	-	-
6	AAS	12.5 $\pm$ 1.40	204 $\pm$ 29	-	-	-
7	XESM	-	11 $\pm$ (40%)	-	3.9 $\pm$ (50%)	30 $\pm$ (50%)
8	AAS	-	-	-	-	-
9	AAS	3.12 $\pm$ (10%)	42.7 $\pm$ (10%)	-	-	-
10	AAS	4.4 $\pm$ 0.2	27 $\pm$ 1	-	-	-
11	AAS	3.53 $\pm$ 0.07	-	-	-	-
12	AAS	3.2 $\pm$ 0.74	32.4 $\pm$ 8.4	-	-	-
13	AAS	3.7 $\pm$ 0.1	30.4 $\pm$ 0.9	-	-	-
14	AAS	4.8 $\pm$ 0.3	34	3.8 $\pm$ 0.5	-	-
	ASV	-	-	-	-	-
15	AAS	4.02 $\pm$ (5.1%)	38.1 $\pm$ (3.8%)	2.10 $\pm$ (2.8%)	3.28 $\pm$ (3.6%)	-
16	AAS	6.5 $\pm$ 0.6	31.6 $\pm$ 1.3	-	-	-
	NAA	3.8 $\pm$ 0.3	34.1 $\pm$ 1.13	-	-	-
17	NAA	-	46.6 $\pm$ (6.6%)	-	2.22 $\pm$ (10.0%)	-
18	AAS	5.000 $\pm$ (5.74%)	36.620 $\pm$ (1.72%)	-	-	-
19	AAS	4.17 $\pm$ (4%)	32.28 $\pm$ (4%)	-	-	-
20	XRF	-	30.0 $\pm$ 5	-	-	-
21	AAS	4.1 $\pm$ (5%)	34.7 $\pm$ (5%)	-	-	-
22	AAS	-	32.2 $\pm$ 0.4	-	-	-
23	AAS	3.9 $\pm$ (9%)	33.8 $\pm$ (2%)	-	-	0.13 $\pm$ (62%)
24	AAS	6.08 $\pm$ 0.49	32.3 $\pm$ 1.13	-	-	-
25	XESM	5.6 $\pm$ 0.2	37.7 $\pm$ 0.9	2.2 $\pm$ 0.9	1.2 $\pm$ 0.2	-
26	AAS	4.03 $\pm$ (5.5%)	34.5 $\pm$ (2.2%)	-	-	-
27	AAS	4.12 $\pm$ 0.76	33.1 $\pm$ 1.0	-	-	-
28	AAS	17.6 $\pm$ (5.7%)	35.6 $\pm$ (1.6%)	-	-	-
	AAS	4.1 $\pm$ (11.4%)	35.5 $\pm$ (2.6%)	-	-	-
29	AAS	3.1	26.1	-	-	-
30	XRF	-	30 $\pm$ 4	-	-	-

TABLE 2 (continued)

Code No.	Method	Cu	Zn	As	Se	Ag
31	NAA	3.96 ± 0.05	29.9 ± 1.1	2.98 ± 0.11	-	-
32	AAS	3.81 ± 0.186	51.64 ± 2.14	-	-	-
33	AAS	-	20.288 ± (10.47%)	-	-	-
34	AAS	4.25 ± (3%)	33.3 ± (2%)	-	-	-
35	NAA	8.8 ± (10%)	31.4 ± (3.5%)	2.1 ± (2.8%)	1.28 ± (5%)	-
36	AAS	4.60 ± (5%)	33.08 ± (5%)	-	-	-
37	AAS	4.38 ± (3%)	28.6 ± (10%)	-	-	-
38	AAS	5.1 ± (10%)	17.0 ± (4%)	-	-	-
39	NAA	-	38.6 ± (11.1%)	-	1.37 ± (11.5%)	-
40	AAS	0.578 ± (13.7%)	36.90 ± (18.3%)	-	-	-
41	NAA	3.9 ± 0.2	-	2.02 ± 0.05	-	-
42	AAS	4.4 ± (5%)	35.7 ± (5%)	-	-	-
43	AAS	2.9 ± (10%)	33 ± (5%)	-	-	0.08 ± (20%)
	NAA	4.1 ± (10%)	34 ± (10%)	-	-	0.12 ± (20%)
44	AAS	-	-	-	-	-
45	AAS	3.32	34.5	-	-	-
46	AAS	3.94 ± (5%)	34.2 ± (5%)	-	-	-
	NAA	-	29.9 ± 0.71	3.18 ± 0.12	3.92 ± 0.07	0.12 ± 0.02
47	NAA	-	37.5 ± (1.7%)	3.05 ± (2.1%)	1.39 ± (5.0%)	0.081 ± (28.3%)
48	NAA	-	-	2.25 ± (10%)	-	-
49	AAS	-	38.8 ± 0.3	-	-	-
	NAA	-	41.7 ± 1.5	2.95 ± 0.25	1.20 ± 0.12	-
50	AAS	-	-	-	-	-
51	AAS	3.8 ± 0.8	28 ± 4	-	-	-
	NAA	-	29 ± 3	-	-	-
	SSMS	3 ± (30%)	-	0.75 ± (30%)	0.5 ± (30%)	<0.1 ± (30%)
52	AAS	4.42 ± 0.21	31.95 ± 1.80	-	-	0.42 ± 0.03
53	AAS	3.98 ± 0.15	33.89 ± 0.80	-	-	-
54	AAS	4.0 ± 0.2	34 ± 1	-	-	0.23 ± 0.08
55	NAA	<20	34.4 ± 0.5	2.91 ± 0.06	1.18 ± 0.01	0.091 ± 0.006
	XRF	3.5 ± 0.3	32.9 ± 0.6	2.7 ± 0.2	1.07 ± 0.07	-
56	AAS	6.92 ± 1.10	29.3 ± 1.0	-	-	-

TABLE 3
RESULT OF TRACE ELEMENT MEASUREMENTS ON THE FISH FLESH SAMPLE MA-A-2
(Cd, Sb, Hg, Pb $\mu\text{g/g}$ -dry matter)

Code No.	Method	Cd	Sb	Hg	Pb
1	AAS	0.074 $\pm$ (8.2%)	-	0.768 $\pm$ (2.4%)	0.199 $\pm$ (8.0%)
2	AAS	-	-	0.62 $\pm$ 0.02	-
	ASV	0.02 $\pm$ 0.01	-	-	0.42 $\pm$ 0.06
3	AAS	< 0.3 $\pm$ (10%)	-	0.56 $\pm$ (10%)	< 1.6 $\pm$ (15%)
4	AAS	-	-	0.57 $\pm$ 0.03	-
5	AAS	0.076 $\pm$ (12%)	-	0.67 $\pm$ (8%)	0.46 $\pm$ (12%)
6	AAS	0.83 $\pm$ 0.14	-	0.33 $\pm$ 0.03	0.31 $\pm$ 0.07
7	XESM	-	-	-	-
8	AAS	-	-	0.39 $\pm$ 0.02	-
9	AAS	0.096 $\pm$ (10%)	-	0.45 $\pm$ (10%)	0.45 $\pm$ (20%)
10	AAS	0.07 $\pm$ 0.01	-	0.523 $\pm$ 0.003	-
11	AAS	0.046 $\pm$ 0.001	-	0.569 $\pm$ 0.005	0.254 $\pm$ 0.020
12	AAS	0.088 $\pm$ 0.081	-	0.57 $\pm$ 0.16	1.8
13	AAS	0.02 $\pm$ 0.01	-	0.509 $\pm$ 0.056	0.3 $\pm$ 0.1
14	AAS	-	-	0.60 $\pm$ 0.05	-
	ASV	0.065 $\pm$ 0.001	-	-	0.39 $\pm$ 0.01
15	AAS	0.09 $\pm$ (33.5%)	-	0.44 $\pm$ (2.2%)	0.29 $\pm$ (15.2%)
16	AAS	-	-	-	-
	NAA	-	-	-	-
17	NAA	-	-	-	-
18	AAS	0.060 $\pm$ (26.35%)	-	0.632 $\pm$ (4.93%)	< 0.500
19	AAS	0.05 $\pm$ (45%)	-	0.45 $\pm$ (11%)	0.31 $\pm$ (29%)
20	XRF	-	-	-	< 2
21	AAS	0.075 $\pm$ 0.005	-	-	0.35 $\pm$ 0.04
22	AAS	-	-	-	-
23	AAS	0.25 $\pm$ (55%)	-	0.516 $\pm$ (14%)	2.7 $\pm$ (30%)
24	AAS	0.271 $\pm$ 0.025	-	0.179 $\pm$ 0.008	0.097 $\pm$ 0.009
25	XESM	-	-	-	2.8 $\pm$ 0.7
26	AAS	0.11 $\pm$ (9.1%)	-	0.58 $\pm$ (3.4%)	0.67 $\pm$ (3.0%)
27	AAS	< 0.13	-	0.48 $\pm$ 0.01	-
28	AAS	-	-	-	-
29	AAS	0.14	-	0.45	0.48
30	XRF	-	-	-	-

TABLE 3 (continued)

Code No.	Method	Cd	Sb	Hg	Pb
31	NAA	0.065 $\pm$ 0.007	0.0038 $\pm$ 0.0003	0.556 $\pm$ 0.020	-
32	AAS	0.168 $\pm$ 0.033	-	-	0.978 $\pm$ 0.31
33	AAS	0.114 $\pm$ (19.32%)	-	0.320 $\pm$ (12.06%)	-
34	AAS	0.086 $\pm$ (5%)	-	-	0.59 $\pm$ (3%)
35	NAA	-	0.14 $\pm$ (17%)	-	-
36	AAS	0.74 $\pm$ (10%)	-	-	1.22 $\pm$ (10%)
37	AAS	<0.1	-	0.208 $\pm$ (10%)	<0.4
38	AAS	0.039 $\pm$ (12%)	-	-	-
39	NAA	-	-	0.54 $\pm$ (10.1%)	-
40	AAS	0.158 $\pm$ (4.8%)	-	0.247 $\pm$ (5.9%)	0.208 $\pm$ (23.3%)
41	NAA	-	-	0.39 $\pm$ 0.03	-
42	AAS	<0.45 $\pm$ (5%)	-	-	<0.4 $\pm$ (10%)
43	AAS	0.05 $\pm$ (20%)	-	0.71 $\pm$ (10%)	0.3 $\pm$ (20%)
	NAA	-	0.004 $\pm$ (50%)	-	-
44	AAS	-	-	0.578 $\pm$ (0.7%)	-
45	AAS	-	-	-	-
46	AAS	$\leq$ 0.32	-	-	$\leq$ 3
	NAA	-	$\leq$ 0.01	0.32 $\pm$ 0.04	-
47	NAA	-	0.053 $\pm$ (12.0%)	0.464 $\pm$ (11.3%)	-
48	NAA	-	-	0.20 $\pm$ (10%)	-
49	AAS	-	-	-	-
	NAA	-	0.0032 $\pm$ (20%)	0.275	-
50	AAS	-	-	0.26	-
51	AAS	$\leq$ 0.06	-	-	$\leq$ 0.5
	NAA	-	-	-	-
	SSMS	<0.2 $\pm$ (30%)	<0.15 $\pm$ (30%)	-	<0.1 $\pm$ (30%)
52	AAS	0.44 $\pm$ 0.17	-	0.58 $\pm$ 0.02	1.49 $\pm$ 0.27
53	AAS	-	-	0.55 $\pm$ 0.05	0.88 $\pm$ 0.05
54	AAS	-	-	0.60 $\pm$ 0.03	-
55	NAA	-	0.010 $\pm$ 0.001	0.37 $\pm$ 0.01	-
	XRF	<2	<3	<1	<1
56	AAS	-	-	-	-

TABLE 4 PRELIMINARY OVERALL AVERAGE VALUES FOR THE RESULTS OF TRACE
ELEMENT MEASUREMENTS REPORTED ON FISH FLESH SAMPLE WA-A-2
(30 September 1978)

Element	No. of values reported	Overall Average ($\mu\text{g/g-dry}$)
Cr	25	2.9 ± 0.6 (21%)
Mn	28	1.0 ± 0.1 (10%)
Fe	35	61 ± 4 (6.6%)
Co	17	0.4 ± 0.2 (50%)
Ni	18	1.2 ± 0.2 (17%)
Cu	44	4.6 ± 0.4 (8.7%)
Zn	53	36 ± 3 (8.3%)
As	12	2.5 ± 0.2 (8.0%)
Se	12	1.9 ± 0.3 (16%)
Ag	8	4 ± 4 (100%)
Cd	27	0.16 ± 0.04 (25%)
Sb	5	0.04 ± 0.03 (75%)
Hg	37	0.48 ± 0.02 (4.2%)
Pb	24	0.7 ± 0.2 (29%)

The uncertainties given are standard errors.

ANNEX

LIST OF LABORATORIES WHICH REPORTED THE RESULTS
(30 September 1978)

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PROGRESS REPORT No. 19
INTERCALIBRATION OF ANALYTICAL METHODS
ON MARINE ENVIRONMENTAL SAMPLES

International Laboratory of Marine Radioactivity
International Atomic Energy Agency
Musée Océanographique
Principality of Monaco

November 1978

Trace element measurements on Sea Plant (SP-M-1) and Copepod (MA-A-1) (Supplement)

The results of the preliminary survey, made on the trace element measurement data reported for sea plant (SP-M-1) and copepod (MA-A-1) before June 1977, have already been presented in Progress Report No. 15 (PR-15). Since that time, further results have been received from a number of laboratories. Altogether 132 laboratories received the sea plant and/or the copepod sample(s). As of end of September 1978, 95 laboratories including those whose data had already been compiled in PR-15 reported results. The present report gives the result of a survey made on the reported data, including those received after the publication of PR-15.

Only individual results reported from 43 laboratories after June 1977 on the 14 selected trace elements (as selected in PR-15) are given here as supplements. Tables 1 and 4 (Cr, Mn, Fe, Co and Ni), Tables 2 and 5 (Cu, Zn, As, Se and Ag) and Tables 3 and 6 (Cd, Sb, Hg and Pb) represent the supplements to the corresponding tables in PR-15.

These additional 43 laboratories are listed in the Annex. The annex also includes a list of the laboratories which declined to report (30 September 1978).

The abbreviations used in Tables 1-6 (supplement) are:

AAS: Atomic Absorption Spectrophotometry	NAA (chem): NAA with chemical separation
ASV: Anodic Stripping Voltammetry	NAA (inst): NAA Instrumental
ESM: Emission Spectrometry	SPM: Spectrophotometry
MAS: Molecular Absorption Spectrometry	XRF: X-Ray Fluorescence Spectrometry
NAA: Neutron Activation Analysis	SSMS: Spark Source Mass Spectrometry

N.D.: Not detected or not determined due to too low concentration.

Not Partic.: Not Participated

The procedures adopted for compiling individual data are similar to those described in PR-15.

Tables 7, 8 and 9 show ranges and overall averages of all values reported for the 14 selected elements in the sea plant sample (SP-M-1); Tables 10, 11 and 12 are of the copepod sample (MA-A-1). In these tables, average values, obtained after rejecting outliers by applying Chauvenet's or Dixon's test, are also given.

Based on the standard errors associated with these average values computed after the rejection of outliers, the values for the following elements are considered to be close to the "true concentrations":

SP-M-1: Mn, Fe, Ni, Cu, Zn

MA-A-1: Fe, Cu, Zn, Cd, Hg

For the remaining elements, there is no guarantee that these average values are close to the "true concentrations", even though they may not be far off.

Especially the values for the following elements have to be looked at with caution:

SP-M-1: Cr, Co, Ag, Cd, Sb, Pb

MA-A-1: Cr, Co, Ni, Ag, Sb, Pb

TABLE 1 RESULTS OF TRACE ELEMENT MEASUREMENTS ON THE SEA PLANT SAMPLE SP-M-1
(Cr, Mn, Fe, Co, Ni $\mu\text{g/g}$ -dry matter)
(SUPPLEMENT)

Code No.	Method	Cr	Mn	Fe	Co	Ni
53	NAA	7.50 $\pm$ (7%)	57.7 $\pm$ (4%)	1910 $\pm$ (7%)	2.58 $\pm$ (7%)	-
54	NAA(inst)	-	-	1900 $\pm$ 200	-	-
	NAA(chem)	4.2 $\pm$ 0.2	-	1800 $\pm$ 200	2.4 $\pm$ 0.1	20 $\pm$ 3
55	AAS	-	67.0 $\pm$ (7.9%)	-	-	-
56	AAS	4.65 $\pm$ 0.50	-	1659 $\pm$ 52.60	13.36 $\pm$ 1.59	16.98 $\pm$ 1.88
57	AAS	-	-	-	-	-
58	AAS	6	57 $\pm$ (20%)	-	6 $\pm$ (40%)	27 $\pm$ (20%)
	XRF	-	-	2170 $\pm$ (10%)	-	-
	SSMS	-	45 $\pm$ (20%)	-	-	-
59	AAS	-	77.3 $\pm$ (8%)	-	-	-
60	AAS	5.6 $\pm$ 0.3	-	1748 $\pm$ 198.7	-	-
61	AAS	-	-	-	-	-
62	AAS	-	-	-	-	-
	ASV	-	-	-	-	-
63	NAA	4.12 $\pm$ 0.44	60.2 $\pm$ 4.0	1833 $\pm$ 126	3.96 $\pm$ 1.08	18.1 $\pm$ 1.1
64A	AAS	-	59.85 $\pm$ (0.69%)	-	-	23.12 $\pm$ (0.74%)
64B	AAS	-	-	-	-	-
64C	AAS	-	64.66	445.33	-	-
65	AAS	-	-	-	-	-
66	Not Partic.	-	-	-	-	-
67	XRF	7.5 $\pm$ 1	-	-	-	-
68	AAS	22.01 $\pm$ (10%)	54.27 $\pm$ (5%)	1395.0 $\pm$ (5%)	17.12 $\pm$ (5%)	6.55 $\pm$ (10%)
69	-	-	-	-	-	-
70	AAS	-	55 $\pm$ ($\leq$ 5%)	1980 $\pm$ ($\leq$ 5%)	2.9 $\pm$ ($\leq$ 5%)	19.4 $\pm$ ($\leq$ 5%)
71	AAS	2.7 $\pm$ (22%)	48 $\pm$ (7%)	2000 $\pm$ (8%)	1.9 $\pm$ (21%)	15.2 $\pm$ (13%)
72	NAA	4.2 $\pm$ (10%)	-	-	2.2 $\pm$ (5%)	-
73	NAA	6.4 $\pm$ 0.3	-	-	2.9 $\pm$ 0.1	-
74	AAS	3.8	60.67	1697	1.73	14.53
	MAS	-	-	-	-	-
75	Not Partic.	-	-	-	-	-

TABLE 1 (continued)

Code No.	Method	Cr	Mn	Fe	Co	Ni
76	AAS	-	-	-	-	14.85 $\pm$ 0.91
77	AAS	-	-	-	-	16.09 $\pm$ 0.24
	ASV	-	-	-	-	-
78	AAS	-	-	-	-	-
79	AAS	-	-	-	-	-
80	NAA(inst) 3.1 $\pm$ (10%)	-	-	1680 $\pm$ (5%)	2.18 $\pm$ (10%)	-
	NAA(chem)	-	-	-	-	-
81	NAA	-	-	-	-	-
82	ESM 15.0	-	-	-	< 4.2	21.9
83	AAS 2.9 $\pm$ (3%)	48.0 $\pm$ (2%)	1171 $\pm$ (2%)	1.9 $\pm$ (17%)	15.8 $\pm$ (2%)	-
84	- <0.1	41	1630	0.9	8.7	-
85	AAS	63.2 $\pm$ (5%)	1961 $\pm$ (5%)	-	-	-
86A	AAS	63 $\pm$ 5	1660 $\pm$ 25	29 $\pm$ 5	42 $\pm$ 5	-
86B	AAS 10.7 $\pm$ 0.8	58 $\pm$ 7	1700 $\pm$ 50	-	23 $\pm$ 9	-
86C	AAS	66 $\pm$ 3	1810 $\pm$ 30	24 $\pm$ 6	38 $\pm$ 3	-
87	XRF	48 $\pm$ 8	1150 $\pm$ 170	-	17 $\pm$ 4	-
88	XRF <6	56 $\pm$ (15%)	2170 $\pm$ (12%)	-	21 $\pm$ (15%)	-
89	AAS 2.5	55.1 $\pm$ 0.7	1794 $\pm$ 253	<4.9	15 $\pm$ 1.7	-
	SPM	-	-	-	-	-
90	AAS	78.6 $\pm$ 2.1	2030 $\pm$ 46	-	-	-
91	AAS	-	-	-	-	-
92	AAS	-	-	-	-	-
93	AAS	-	-	-	-	-
94	AAS	-	-	-	-	-
95	AAS	64.6 $\pm$ (3.7%)	-	-	-	-
	AAS	64.0 $\pm$ (4.1%)	-	-	-	-

TABLE 2 RESULTS OF TRACE ELEMENT MEASUREMENTS ON THE SEA PLANT SAMPLE SP-14-1
(Cu, Zn, As, Se, Ag $\mu\text{g/g}$ -dry matter)
(SUPPLEMENT)

Code No.	Method	Cu	Zn	As	Se	Ag
53	NAA	-	81 $\pm$ (7%)	-	-	-
54	NAA(inst)	-	65 $\pm$ 8	-	-	-
	NAA(chem)	11.2 $\pm$ 0.6	66 $\pm$ 5	3.2 $\pm$ 0.2	0.19 $\pm$ 0.02	-
55	AAS	-	94.6 $\pm$ (2.1%)	-	-	-
56	AAS	3.30 $\pm$ 0.112	63.67 $\pm$ 1.01	-	-	-
57	AAS	-	-	-	-	-
58	AAS	16 $\pm$ (20%)	64 $\pm$ (15%)	-	-	1.7 $\pm$ (30%)
	XRF	-	-	-	-	-
	SSMS	18.6 $\pm$ (20%)	-	4.9 $\pm$ (20%)	-	1.3 $\pm$ (20%)
59	AAS	11.5 $\pm$ (5%)	81.3 $\pm$ (8%)	-	-	-
60	AAS	13.2 $\pm$ 1.13	-	-	-	-
61	AAS	9.6 $\pm$ (5.7%)	-	-	-	1.93 $\pm$ (15%)
62	AAS	10.2 $\pm$ 1.0	54.3 $\pm$ 2.0	-	-	-
	ASV	-	-	-	-	-
63	NAA	-	69.9 $\pm$ 4.5	-	0.12 $\pm$ 0.03	-
64A	AAS	13.50 $\pm$ (0.52%)	57.51 $\pm$ (1.03%)	-	-	1.25 $\pm$ (3.73%)
64B	AAS	-	-	-	-	-
64C	AAS	-	44.34	-	-	-
65	AAS	-	-	-	-	-
66	AAS	-	-	-	-	-
67	XRF	17 $\pm$ 3	65 $\pm$ 5	-	-	-
68	AAS	9.24 $\pm$ (5%)	51.24 $\pm$ (5%)	-	-	-
69	-	-	-	-	-	-
70	AAS	12.9 $\pm$ (<5%)	59.4 $\pm$ (<5%)	-	-	-
71	AAS	22 $\pm$ (24%)	73 $\pm$ (11%)	-	-	-
72	NAA	-	-	2.61 $\pm$ (88%)	-	-
73	NAA	-	-	-	-	-
74	AAS	10.8	58	-	-	-
	MAS	-	-	0.4	-	-
75	Not Partic.	-	-	-	-	-

TABLE 2 (continued)

Code No.	Method	Cu	Zn	As	Se	Ag
76	AAS	11.7 $\pm$ 0.22	66.0 $\pm$ 0.29	-	-	-
77	AAS	13.6	-	-	-	-
	ASV	9.32	-	-	-	-
78	AAS	-	-	-	-	-
79	AAS	-	-	-	-	-
80	NAA(inst)	-	78 $\pm$ (10%)	-	-	-
	NAA(chem)	-	-	2.55 $\pm$ (5%)	0.114 $\pm$ (10%)	-
81	NAA	-	-	-	-	-
82	ESM	9.0	-	-	-	-
83	AAS	11.2 $\pm$ (1%)	60.9 $\pm$ (2%)	-	-	-
84		10.2	37	<1		<1
85	AAS	14.7 $\pm$ (5%)	66.2 $\pm$ (5%)	-	-	-
86A	AAS	17 $\pm$ 1	70 $\pm$ 2	-	-	-
86B	AAS	-	-	-	-	-
86C	AAS	13 $\pm$ 1	60 $\pm$ 2	-	-	-
87	XRF	14 $\pm$ 3	55 $\pm$ 9	13 $\pm$ 3	3 $\pm$ 1	-
88	XRF	13 $\pm$ (20%)	67 $\pm$ (12%)	2.8 $\pm$ (20%)	<2	-
89	AAS	13.7 $\pm$ 3.1	53.3 $\pm$ 1.8	-	-	1.2
	SPM	-	-	3.4 $\pm$ 0.14	-	-
90	AAS	12.1 $\pm$ 0.2	62.6 $\pm$ 2.1	-	-	-
91	AAS	-	1.584 $\pm$ (11.11%)	-	-	-
92	AAS	-	-	-	-	-
93	AAS	-	-	-	-	-
94	AAS	13.39 $\pm$ 0.27	67.06 $\pm$ 0.33	-	-	-
95	AAS	21.4 $\pm$ (7.3%)	108.0 $\pm$ (8.6%)	-	-	-
	AAS	10.5 $\pm$ (1.6%)	100.7 $\pm$ (1.7%)	-	-	-

TABLE 3 RESULTS OF TRACE ELEMENT MEASUREMENTS ON THE SEA PLANT SAMPLE SP-M-1
(Cd, Sb, Hg, Pb $\mu\text{g/g}$ -dry matter)
(SUPPLEMENT)

Code No.	Method	Cd	Sb	Hg	Pb
53	NAA	-	0.53 $\pm$ (12%)	-	-
54	NAA(inst)	-	-	-	-
	NAA(chem)	0.35 $\pm$ 0.03	0.49 $\pm$ 0.08	0.34 $\pm$ 0.03	-
55	AAS	0.43 $\pm$ (8.1%)	-	0.36 $\pm$ (7.7%)	-
56	AAS	0.251 $\pm$ 0.014	-	0.289 $\pm$ 0.023	1.29 $\pm$ 0.08
57	AAS	0.20 $\pm$ 0.03	-	0.57 $\pm$ 0.06	15.7 $\pm$ 1.3
58	AAS	1 $\pm$ (50%)	-	-	2.2 $\pm$ (30%)
	XRF	-	-	-	-
	SSMS	-	-	-	21.1 $\pm$ (20%)
59	AAS	1.09 $\pm$ (6%)	-	0.357 $\pm$ (6%)	28.1 $\pm$ (8%)
60	AAS	0.18 $\pm$ 0.01	-	-	10.5 $\pm$ 2.25
61	AAS	0.44 $\pm$ (10.8%)	-	0.223 $\pm$ (15.2%)	11.9 $\pm$ (6.4%)
62	AAS	0.43 $\pm$ 0.05	-	0.18 $\pm$ 0.04	-
	ASV	-	-	-	15.2 $\pm$ 2.0
63	NAA	-	0.50 $\pm$ 0.04	0.85	-
64A	AAS	-	-	-	23.79 $\pm$ (1.05%)
64B	AAS	-	-	0.053 $\pm$ 0.005	24.13 $\pm$ 0.13
64C	AAS	3.42	-	0.05	45.78
65	AAS	-	-	0.4765	-
66	AAS	-	-	-	-
67	XRF	-	-	-	-
68	AAS	-	-	-	19.60 $\pm$ (10%)
69	-	-	-	-	-
70	AAS	0.36 $\pm$ ($<$ 5%)	-	-	15.8 $\pm$ ($<$ 5%)
71	AAS	0.40 $\pm$ (20%)	-	-	0.72 $\pm$ (28%)
72	NAA	-	1.3 $\pm$ (32%)	-	-
73	NAA	-	-	-	-
74	AAS	0.4	-	0.38	17.27
	MAS	-	-	-	-
75	Not Partic.	-	-	-	-

TABLE 3 (continued)

Code No.	Method	Cd	Sb	Hg	Pb
76	AAS	-	-	0.347 $\pm$ 0.032	14.7 $\pm$ 0.78
77	AAS	0.483	-	0.336 $\pm$ 0.140	17.5
	ASV	0.306	-	-	14.6
78	AAS	0.69	-	0.27	-
79	AAS	-	-	0.190 $\pm$ 0.032	-
80	NAA(inst)	-	-	-	-
	NAA(chem)	-	0.30 $\pm$ (10%)	0.31 $\pm$ (5%)	-
81	NAA	-	-	-	-
82	ISM	-	-	-	-
83	AAS	0.31 $\pm$ (14%)	-	-	16.8 $\pm$ (6%)
84	-	<1	-	0.444	6
85	AAS	-	-	-	-
86A	AAS	-	-	-	-
86B	AAS	0.23 $\pm$ 0.05	-	0.40 $\pm$ 0.03	-
86C	AAS	-	-	0.210 $\pm$ 0.030	-
87	XRF	-	-	-	-
88	XRF	-	-	-	28 $\pm$ (15%)
89	AAS	<1.2	-	0.241 $\pm$ 0.020	12 $\pm$ 0
	SPM	-	-	-	-
90	AAS	-	-	0.310 $\pm$ 0.015	-
	AAS	-	-	0.362 $\pm$ 0.009	-
91	AAS	0.953 $\pm$ (6.5%)	-	0.661 $\pm$ (4.36%)	-
92	AAS	-	-	0.249 $\pm$ 0.0022	-
93	AAS	1.25 $\pm$ 0.06	-	0.41 $\pm$ 0.05	13.5 $\pm$ 3.0
94	AAS	-	-	-	-
95	AAS	-	-	-	-

TABLE 4 RESULTS OF TRACE ELEMENT MEASUREMENTS ON THE COPEPOD SAMPLE MA-A-1
(Cr, Mn, Fe, Co, Ni $\mu\text{g/g}$ dry matter)
(SUPPLEMENT)

Code No.	Method	Cr	Mn	Fe	Co	Ni
53	NAA	2.59 $\pm$ (5%)	-	66 $\pm$ (7%)	0.18 $\pm$ (5%)	-
54	NAA(inst)	-	<10	-	-	-
	NAA(chem)	0.23 $\pm$ 0.03	-	59 $\pm$ 9	0.12 $\pm$ 0.01	1.5 $\pm$ 0.3
55	AAS	-	3.00 $\pm$ (6%)	-	-	-
56	AAS	2.57 $\pm$ 0.34	-	58 $\pm$ 2.3	1.27 $\pm$ 0.19	2.52 $\pm$ 0.34
57	AAS	-	-	-	-	-
58	Not Partic.	-	-	-	-	-
59	AAS	-	3.93 $\pm$ (8%)	-	-	-
60	AAS	1.43 $\pm$ 0.38	-	88.2 $\pm$ 13.3	-	-
61	AAS	-	-	69.1 $\pm$ (4.6%)	-	-
62	AAS	-	-	-	-	-
	ASV	-	-	-	-	-
63	NAA	-	-	54.7 $\pm$ 3.5	0.42 $\pm$ 0.07	-
64A	AAS	-	2.90 $\pm$ (1.79%)	-	-	1.618 $\pm$ (5.09%)
64B	AAS	-	-	-	-	-
64C	AAS	-	0.97	35.29	-	-
65	AAS	-	-	-	-	-
66	AAS	0.88	3.11	48.43	-	-
67	XRF	3.5 $\pm$ 0.5	-	62 $\pm$ 5	-	-
68	AAS	2.62 $\pm$ (10%)	3.23 $\pm$ (5%)	38.2 $\pm$ (5%)	6.17 $\pm$ (5%)	N.D.
69	Not Partic.	-	-	-	-	-
70	AAS	-	1.9 $\pm$ (<5%)	40 $\pm$ (<5%)	0.3 $\pm$ (<5%)	1.5 $\pm$ (<5%)
71	AAS	-	-	29 $\pm$ (15%)	0.13 $\pm$ (25%)	-
72	NAA	-	-	-	0.14 $\pm$ (49%)	-
73	NAA	1.5 $\pm$ 0.2	-	-	0.59 $\pm$ 0.04	-
74	AAS	0.8	6.07	76	< 0.2	1.1
	MAS	-	-	-	-	-
75	NAA	-	-	67 $\pm$ 7	0.17 $\pm$ 0.02	-
	AAS	<1	5.2 $\pm$ 0.3	46 $\pm$ 7	6.0	3.2 $\pm$ 0.6

TABLE 4 (continued)

Code No.	Method	Cr	Mn	Fe	Co	Ni
76	AAS	-	-	-	-	0.96 ± 0.04
77	AAS	-	-	-	-	2.91 ± 0.26
	ASV	-	-	-	-	-
78	AAS	-	-	-	-	-
79	AAS	-	-	-	-	-
80	NAA(inst)<0.5	-	-	-	$0.049 \pm (15\%)$	-
	NAA(chem)	-	-	-	-	-
81	NAA	-	-	-	-	-
82	ESM	<1.8	-	-	<1.8	2.9
83	AAS	$0.40 \pm (8\%)$	$3.1 \pm (2\%)$	$78.0 \pm (2\%)$	$0.14 \pm (20\%)$	$1.4 \pm (10\%)$
84	-	<0.1	2.7	49	<0.5	1.7
85	Not Partic.	-	-	-	-	-
86	Not Partic.	-	-	-	-	-
87	XRF	-	-	59 ± 13	-	-
88	XRF	<6	<4	$58 \pm (20\%)$	-	<2
89	AAS	<1.2	2.44 ± 0.24	59.6 ± 2.2	<4.7	<2.4
	SPM	-	-	-	-	-
90	AAS	-	2.49 ± 0.17	58.3 ± 3.0	-	-
91	AAS	-	-	-	-	-
92	AAS	-	-	-	-	-
93	AAS	-	-	-	-	-
94	AAS	-	-	-	-	-
95	AAS	-	$3.8 \pm (10.5\%)$	-	-	-

TABLE 5 RESULTS OF TRACE ELEMENT MEASUREMENTS ON THE COPEPOD SAMPLE MA-A-1
(Cu, Zn, As, Se, Ag $\mu\text{g/g}$ dry matter)
(SUPPLEMENT)

Code No.	Method	Cu	Zn	As	Se	Ag
53	NAA	7.43 $\pm$ (4%)	191 $\pm$ (3%)	-	-	-
54	NAA(inst)	-	160 $\pm$ 13	-	-	-
	NAA(chem)	7.1 $\pm$ 0.4	165 $\pm$ 11	8.2 $\pm$ 0.7	3.4 $\pm$ 0.2	-
55	AAS	9.53 $\pm$ (4.3%)	174 $\pm$ (7.6%)	-	-	-
56	AAS	4.49 $\pm$ 0.19	173 $\pm$ 3.4	-	-	-
57	AAS	-	-	-	-	-
58	Not Partic.	-	-	-	-	-
59	AAS	8.38 $\pm$ (5%)	223 $\pm$ (8%)	-	-	-
60	AAS	7.6 $\pm$ 0.42	-	-	-	-
61	AAS	7.54 $\pm$ (6.2%)	-	-	-	0.44 $\pm$ (22.7%)
62	AAS	5.5 $\pm$ 0.5	151.6 $\pm$ 5.0	-	-	-
63	NAA	-	147 $\pm$ 4.7	-	1.32 $\pm$ 0.06	-
64A	AAS	7.96 $\pm$ (0.29%)	182.54 $\pm$ (1.77%)	-	-	0.447 $\pm$ (13.42%)
64B	AAS	-	-	-	-	-
64C	AAS	-	62.84	-	-	-
65	AAS	-	-	-	-	-
66	AAS	6.74	146.38	1.04	9.4	0.47
67	XRF	9 $\pm$ 2	165 $\pm$ 5	-	-	-
68	AAS	5.99 $\pm$ (5%)	219.71 $\pm$ (5%)	-	-	-
69		-	-	-	-	-
70	AAS	8.2 $\pm$ (<5%)	162 $\pm$ (<5%)	-	-	-
71	AAS	12.5 $\pm$ (30%)	156 $\pm$ (8%)	-	-	-
72	NAA	-	139.4 $\pm$ (9%)	3.11 $\pm$ (45%)	-	-
73	NAA	-	-	-	-	-
74	AAS	7.47	151.7	-	-	-
	MAS	-	-	3.0	-	-
75	NAA	-	133 $\pm$ 13	-	2.8 $\pm$ 0.3	-
	AAS	4.7 $\pm$ 0.4	141 $\pm$ 4	-	-	-

TABLE 5 (continued)

Code No.	Method	Cu	Zn	As	Se	Ag
76	AAS	7.5 $\pm$ 0.15	192.0 $\pm$ 0.81	-	-	-
77	AAS	6.9	-	-	-	-
	ASV	6.93	-	-	-	-
78	AAS	-	-	-	-	-
79	AAS	-	-	-	-	-
80	NAA(inst)	-	-	-	-	-
	NAA(chem)	-	-	7.4 $\pm$ (5%)	2.03 $\pm$ (10%)	-
81	NAA	-	-	-	-	-
82	ESM	4.7	-	-	-	-
83	AAS	7.39	161 $\pm$ (2%)	-	-	-
84	-	6.8	126	<1	-	<1
85	Not Partic.	-	-	-	-	-
86	Not Partic.	-	-	-	-	-
87	XRF	11 $\pm$ 4	153 $\pm$ 31	-	-	-
88	XRF	6.7 $\pm$ (20%)	163 $\pm$ (15%)	4.7 $\pm$ (15%)	4.4 $\pm$ (15%)	-
89	AAS	8.23 $\pm$ 1.42	147 $\pm$ 7	-	-	<1.2
	SPM	-	-	6.14 $\pm$ 0.34	-	-
90	AAS	6.92 $\pm$ 0.23	174 $\pm$ 2.6	-	-	-
91	AAS	-	4.250 $\pm$ (2.9%)	-	-	-
92	AAS	-	-	-	-	-
93	AAS	-	-	-	-	-
94	AAS	7.22 $\pm$ 0.40	172.99 $\pm$ 1.32	-	-	-
95	AAS	16.3 $\pm$ (7.6%)	118.1 $\pm$ (6.5%)	-	-	-

TABLE 6 RESULTS OF TRACE ELEMENT MEASUREMENTS ON THE COPEPOD SAMPLE MA-A-1
(Cd, Sb, Hg, Pb $\mu\text{g/g}$ -dry matter)
(SUPPLEMENT)

Code No.	Method	Cd	Sb	Hg	Pb
53	NAA	0.64 $\pm$ (6%)	-	-	-
54	NAA(inst)	-	-	-	-
	NAA(chem)	0.72 $\pm$ 0.09	0.027 $\pm$ 0.008	0.27 $\pm$ 0.03	-
55	AAS	0.90 $\pm$ (8.8%)	-	0.31 $\pm$ (5%)	0.73 $\pm$ (18.5%)
56	AAS	1.09 $\pm$ 0.08	-	0.4342 $\pm$ 0.034	0.86 $\pm$ 0.07
57	AAS	0.65 $\pm$ 0.07	-	0.32 $\pm$ 0.03	21.0 $\pm$ 1.5
58	Not Partic.	-	-	-	-
59	AAS	0.83 $\pm$ (6%)	-	0.301 $\pm$ (6%)	10.1 $\pm$ (8%)
60	AAS	0.34 $\pm$ 0.071	-	-	1.73 $\pm$ 0.03
61	AAS	0.893 $\pm$ (29.7%)	-	0.258 $\pm$ (16.3%)	0.886 $\pm$ (26.2%)
62	AAS	0.71 $\pm$ 0.07	-	0.31 $\pm$ 0.03	-
	ASV	-	-	-	0.9 $\pm$ 0.2
63	NAA	-	-	-	-
64A	AAS	-	-	-	3.09 $\pm$ (4.02%)
64B	AAS	-	-	0.038 $\pm$ 0.005	3.58 $\pm$ 0.005
64C	AAS	2.56	-	0.12	8.58
65	AAS	-	-	0.5208	-
66	AAS	1.04	-	0.295	5.6
67	XRF	-	-	-	-
68	AAS	-	-	-	4.21 $\pm$ (10%)
69	-	-	-	-	-
70	AAS	0.73 $\pm$ (<5%)	-	-	1.0 $\pm$ (<5%)
71	AAS	0.52 $\pm$ (15%)	-	-	-
72	NAA	-	-	-	-
73	NAA	-	-	-	-
74	AAS	0.6	-	0.42	5
	MAS	-	-	-	-
75	NAA	-	-	0.24 $\pm$ 0.08	-
	AAS	0.52 $\pm$ 0.10	-	0.39 $\pm$ 0.05	≤ 7

TABLE 6 (continued)

Code No.	Method	Cd	Sb	Hg	Pb
76	AAS	-	-	0.235 $\pm$ 0.033	1.2 $\pm$ 0.14
77	AAS	0.67 $\pm$ 0.10	-	0.368 $\pm$ 0.080	0.77
	ASV	-	-	-	0.584
78	AAS	0.98	-	0.10 $\sim$ 0.66	-
79	AAS	-	-	0.195 $\pm$ 0.005	-
80	NAA(inst)	-	-	-	-
	NAA(chem)	-	0.041 $\pm$ (15%)	0.236 $\pm$ (5%)	-
81	NAA	-	-	-	-
82	ESM	-	-	-	-
83	AAS	0.71 $\pm$ (5%)	-	-	2.4 $\pm$ (22%)
84	-	<1	-	0.248	<2
85	Not Partic.	-	-	-	-
86	Not Partic.	-	-	-	-
87	XRF	-	-	-	-
88	XRF	-	-	-	4.3 $\pm$ (15%)
89	AAS	<1.2	-	0.163 $\pm$ 0.017	<4.7
	SPM	-	-	-	-
90	AAS	-	-	0.280 $\pm$ 0.029	-
	AAS	-	-	0.270 $\pm$ 0.020	-
91	AAS	0.454 $\pm$ (2.15%)	-	0.440 $\pm$ (10.56%)	-
92	AAS	-	-	0.129 $\pm$ 0.0074	-
93	AAS	0.71 $\pm$ 0.01	-	0.32 $\pm$ 0.02	2.98 $\pm$ 0.13
94	AAS	-	-	-	-
95	AAS	-	-	-	-

TABLE 7 OVERALL AVERAGES AND AVERAGES AFTER CHAUVENET AND DIXON'S TESTS
FOR THE RESULTS REPORTED FOR Cr, Mn, Fe, Co AND Ni MEASUREMENTS
ON SEA PLANT SAMPLE SP-M-1

Element	Cr	Mn	Fe	Co	Ni
No. of reported results*	43	56	57	36	38
Range reported ($\mu\text{g/g-dry}$)	1 - 22.01	40.3 - 100	445.33 - 3334	0.9 - 29	6.55 - 42
Overall Average** ($\mu\text{g/g-dry}$)	5.2 ± 0.6 (12%)	60 ± 2 (3.3%)	1750 ± 50 (2.9%)	5 ± 1 (20%)	20 ± 1 (5.0%)
<u>Chauvenet's Test</u>					
No. of results accepted by the test	39	53	55	28	34
Average** ($\mu\text{g/g-dry}$)	4.3 ± 0.3 (7.0%)	58 ± 1 (1.7%)	1750 ± 40 (2.3%)	2.3 ± 0.2 (8.7%)	18.6 ± 0.7 (3.8%)
<u>Dixon's Test</u>					
No. of results accepted by the test	42	56	56	32	38
Average** ($\mu\text{g/g-dry}$)	4.8 ± 0.4 (8.3%)	60 ± 2 (3.3%)	1730 ± 40 (2.3%)	2.8 ± 0.3 (11%)	20 ± 1 (5.0%)

* Results showing only detection limits of methods used are not included.

** The standard deviations given are for mean (standard error).

TABLE 8 OVERALL AVERAGES AND AVERAGES AFTER CHAUVENET AND DIXON'S TESTS
FOR THE RESULTS REPORTED FOR Cu, Zn, As, Se AND Ag MEASUREMENTS
ON SEA PLANT SAMPLE SP-M-1

Element	Cu	Zn	As	Se	Ag
No. of reported results*	67	75	21	12	15
Range reported ($\mu\text{g/g-dry}$)	3.30 - 43	1.584 - 225	0.4 - 13	0.114 - 3	0.46 - 8.0
Overall Average** ($\mu\text{g/g-dry}$)	13.0 ± 0.6 (4.6%)	66 ± 3 (4.5%)	4.3 ± 0.6 (14%)	0.4 ± 0.2 (50%)	1.7 ± 0.3 (29%)
<u>Chauvenet's Test</u>					
No. of results accepted by the test	62	70	16	11	13
Average** ($\mu\text{g/g-dry}$)	12.3 ± 0.3 (2.4%)	64 ± 2 (3.1%)	3.3 ± 0.2 (6.1%)	0.16 ± 0.01 (6.3%)	1.2 ± 0.1 (8.3%)
<u>Dixon's Test</u>					
No. of results accepted by the test	66	74	19	11	14
Average** ($\mu\text{g/g-dry}$)	12.6 ± 0.4 (3.2%)	64 ± 2 (3.1%)	3.5 ± 0.3 (8.6%)	0.16 ± 0.01 (6.3%)	1.3 ± 0.2 (15%)

* Results showing only detection limits of the methods used are not included.

** The standard deviations given are 1 σ for mean (standard error).

TABLE 9 OVERALL AVERAGES AND AVERAGES AFTER CHAUVENET AND DIXON'S TESTS
FOR THE RESULTS REPORTED FOR Cd, Sb, Hg AND Pb MEASUREMENTS ON
SEA PLANT SAMPLE SP-M-1

Element	Cd	Sb	Hg	Pb
No. of reported results*	46	15	51	49
Range reported ($\mu\text{g/g-dry}$)	0.113 - 24	0.30 - 1.3	0.05 - 5.6	0.72 - 45.78
Overall Average** ($\mu\text{g/g-dry}$)	1.2 ± 0.5 (42%)	0.65 ± 0.06 (9.2%)	0.4 ± 0.1 (25%)	16 ± 1 (6.3%)
<u>Chauvenet's Test</u>				
No. of results accepted by the test	39	14	48	48
Average** ($\mu\text{g/g-dry}$)	0.46 ± 0.04 (8.7%)	0.60 ± 0.05 (8.3%)	0.32 ± 0.02 (6.3%)	16 ± 1 (6.3%)
<u>Dixon's Test</u>				
No. of results accepted by the test	45	15	50	49
Average** ($\mu\text{g/g-dry}$)	0.7 ± 0.1 (14%)	0.65 ± 0.06 (9.2%)	0.33 ± 0.02 (6.1%)	16 ± 1 (6.3%)

* Results showing only detection limits of the methods used are not included.

** The standard deviations given are for mean (standard error).

TABLE 10 OVERALL AVERAGES AND AVERAGES AFTER CHAUVENET AND DIXON'S TESTS
FOR THE RESULTS REPORTED FOR Cr, Mn, Fe, Co AND Ni MEASUREMENTS
ON COPEPOD SAMPLE MA-A-1

Element	Cr	Mn	Fe	Co	Ni
No. of reported results*	21	34	46	22	22
Range reported ($\mu\text{g/g-dry}$)	0.083 - 6.90	0.83 - 9.3	29 - 134	0.041 - 6.17	0.96 - 4.3
Overall Average** ($\mu\text{g/g-dry}$)	1.7 ± 0.4 (24%)	3.2 ± 0.3 (9.4%)	61 ± 3 (4.9%)	0.9 ± 0.4 (44%)	2.0 ± 0.2 (10%)
<u>Chauvenet's Test</u>					
No. of results accepted by the test	18	31	45	14	21
Average** ($\mu\text{g/g-dry}$)	1.1 ± 0.2 (18%)	2.9 ± 0.2 (6.9%)	60 ± 2 (3.3%)	0.12 ± 0.01 (8.3%)	1.9 ± 0.2 (11%)
<u>Dixon's Test</u>					
No. of results accepted by the test	19	33	45	18	22
Average** ($\mu\text{g/g-dry}$)	1.2 ± 0.2 (17%)	3.0 ± 0.2 (6.7%)	60 ± 2 (3.3%)	0.20 ± 0.04 (20%)	2.0 ± 0.2 (10%)

* Results showing only detection limits of the methods used are not included.

** The standard deviations given are for mean (standard error).

TABLE 11 OVERALL AVERAGES AND AVERAGES AFTER CHAUVENET AND DIXON'S TESTS
FOR THE RESULTS REPORTED FOR Cu, Zn, As, Se AND Ag MEASUREMENTS
ON COPEPOD SAMPLE MA-A-1

Element	Cu	Zn	As	Se	Ag
No. of reported results*	56	66	19	15	12
Range reported ($\mu\text{g/g-dry}$)	4.49 - 16.3	4.250 - 246.77	1.04 - 11.02	1.32 - 9.4	0.07 - 1.48
Overall Average** ($\mu\text{g/g-dry}$)	7.8 ± 0.3 (3.8%)	157 ± 4 (2.5%)	6.7 ± 0.6 (9.0%)	3.4 ± 0.5 (15%)	0.5 ± 0.1 (20%)
<u>Chauvenet's Test</u>					
No. of results accepted by the test	54	60	19	14	10
Average** ($\mu\text{g/g-dry}$)	7.6 ± 0.2 (2.6%)	158 ± 2 (1.3%)	6.7 ± 0.6 (9.0%)	3.0 ± 0.2 (6.7%)	0.33 ± 0.06 (18%)
<u>Dixon's Test</u>					
No. of results accepted by the test	55	65	19	14	11
Average** ($\mu\text{g/g-dry}$)	7.7 ± 0.2 (2.6%)	159 ± 3 (1.9%)	6.7 ± 0.6 (9.0%)	3.0 ± 0.2 (6.7%)	0.38 ± 0.08 (21%)

* Results showing only detection limits of the methods used are not included.

** The standard deviations given are 1 σ for mean (standard error).

TABLE 12 OVERALL AVERAGES AND AVERAGES AFTER CHAUVENET AND DIXON'S TESTS
FOR THE RESULTS REPORTED FOR Cd, Sb, Hg AND Pb MEASUREMENTS ON
COPEPOD SAMPLE MA-A-1

Element	Cd	Sb	Hg	Pb
No. of reported results *	43	8	43	35
Range reported ($\mu\text{g/g-dry}$)	0.34 - 3.01	0.012 - 0.19	0.038 - 3.6	0.584 - 21.0
Overall Average ** ($\mu\text{g/g-dry}$)	0.85 ± 0.07 (8.2%)	0.07 ± 0.03 (43%)	0.35 ± 0.08 (23%)	3.8 ± 0.8 (21%)
<u>Chauvenet's Test</u>				
No. of results accepted by the test	41	8	40	30
Average ** ($\mu\text{g/g-dry}$)	0.75 ± 0.03 (4.0%)	0.07 ± 0.03 (43%)	0.28 ± 0.01 (3.6%)	2.1 ± 0.3 (14%)
<u>Dixon's Test</u>				
No. of results accepted by the test	41	8	42	33
Average ** ($\mu\text{g/g-dry}$)	0.75 ± 0.03 (4.0%)	0.07 ± 0.03 (43%)	0.27 ± 0.02 (7.4%)	2.8 ± 0.5 (18%)

* Results showing only detection limits of the methods used are not included.

** The standard deviations given are 1 σ for mean (standard error).

ANNEX

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PROGRESS REPORT No. 20
INTERCALIBRATION OF ANALYTICAL METHODS
ON MARINE ENVIRONMENTAL SAMPLES

International Laboratory of Marine Radioactivity, IAEA
Musée Océanographique
Principality of Monaco

April 1980

Trace Element Measurements on Fish-flesh Sample (MA-A-2) (Supplement)

The preliminary survey of the analytical results reported before October 1978 on the fish sample (MA-A-2) has already been conducted and its results were presented in the Progress Report No. 18 (PR-18). At that time the survey was carried out on the results reported by 56 participating laboratories. Since then 25 additional laboratories reported their data by the end of March 1980. Thus, among 116 laboratories which received the sample, 81 sent in the results up to now, while 18 declined to report. Although the results of 25 additional laboratories are available at present, only those of 21 laboratories have been included in the present report owing to the late arrival of the rest for the inclusion in the computations. The individual results reported by these 21 laboratories are listed in Table 1 (Cr, Mn, Fe, Co, Ni), Table 2 (Cu, Zn, As, Se, Ag) and Table 3 (Cd, Sb, Hg, Pb), which supplement the corresponding tables given in PR-18. The abbreviations used in these tables for the analytical methods adopted by the individual laboratories and the procedures employed for compiling individual data are similar to those given in PR-18. A supplementary list of participating laboratories (21 laboratories) is also attached.

Tables 4, 5 and 6 show new ranges and overall averages including the values presented in PR-18 as well as the present report for 14 trace elements. In these tables, the average values, obtained after rejecting outlying results by applying Chauvenet's or Dixon's tests, are also presented.

Based on the standard errors associated with the average values computed after the rejection of outliers, these average values (consensus values) for Mn, Fe, Cu, Zn, As and Hg are considered to be close to the "true concentrations". When the standard errors of the averages are within a range of $\pm 5\%$, past experiences demonstrated that the "consensus values" computed in the manner described above are in good agreement with the "probable concentrations" computed on the basis of the results obtained by reputed laboratories.

The standard errors of the "consensus values" obtained by applying Chauvenet's test and/or Dixon's test for Cr, Co, Ni, Se, Ag, Cd, Sb and Pb exceed $\pm 10\%$. This indicates that the scatter of the reported values is larger for these elements, so that the average values are not guaranteed to be close to the "true concentration", although they may be not very far off. Especially, the average values for Co, Ni, Se, Sb and Pb should be regarded with caution.

TABLE 1 RESULTS OF TRACE ELEMENT MEASUREMENTS ON THE FISH FLESH SAMPLE MA-A-2
(Cr, Mn, Fe, Co, Ni $\mu\text{g/g}$ dry matter)
(SUPPLEMENT)

Code No.	Method	Cr	Mn	Fe	Co	Ni
57	AAS	-	-	-	-	-
58	AAS	-	0.99 ± 0.05	-	-	-
	NAA	4.6 ± 0.79	1.23 ± 0.09	56.9 ± 14.5	0.057 ± 0.11	-
59	AAS	1.7	1.24	50 ± 2	-	<1.0
60	NAA	1.0 ± 0.4	0.6	55.0 ± 7.0	-	-
61	AAS	-	0.85 ± 0.07	66.20 ± 1.15	<1.6	-
62	NAA	1.23 ± 0.13	0.51 ± 0.02	56.2 ± 2.0	0.049 ± 0.003	-
63	AAS	-	-	-	-	-
64	NAA	<1	<1	$57 \pm (10\%)$	$0.090 \pm (10\%)$	-
65	AAS	-	-	-	-	-
66	AAS	<3	<1.5	57.5 ± 2.5	<3	<3
67	AAS	-	-	-	-	-
68	AAS	0.84	1.55	85.5	1.45	-
69	AAS	-	-	18.6	-	-
70	AAS	-	-	32.0	-	-
71	AAS	2.8	3.0	48.4	2.3	-
72	AAS	0.34 ± 0.05	0.91 ± 0.1	52 ± 4	0.095 ± 0.01	1.3 ± 0.2
73	AAS	0.259	-	-	-	0.507
	NAA	-	-	-	-	-
74	DPP	-	-	-	-	-
	ASV	-	-	-	-	-
75	AAS	-	-	-	-	-
76	AAS	-	0.46	34.8	-	-
77	AAS	-	0.86 ± 0.06	43.09 ± 1.52	-	0.65 ± 0.03

TABLE 2 RESULTS OF TRACE ELEMENT MEASUREMENTS ON THE FISH FLESH SAMPLE MA-A-2
(Cu, Zn, As, Se, Ag $\mu\text{g/g}$ -dry matter)
(SUPPLEMENT)

Code No.	Method	Cu	Zn	As	Se	Ag
57	AAS	-	-	-	-	-
58	AAS	9.3	32.7 $\pm$ 2.0	-	-	-
	NAA	10.6 $\pm$ 1.40	31.8 $\pm$ (30%)	7.3 $\pm$ 0.59	11.3 $\pm$ 0.66	-
59	AAS	6.5 $\pm$ 0.2	32 $\pm$ 1	-	-	-
60	NAA	-	-	-	-	-
61	AAS	4.30 $\pm$ 0.30	35.20 $\pm$ 0.29	-	-	-
62	NAA	3.2 $\pm$ 0.2	34.0 $\pm$ 0.4	2.64 $\pm$ 0.12	1.27 $\pm$ 0.07	-
63	AAS	-	-	-	-	-
64	NAA	6.1 $\pm$ (20%)	33 $\pm$ (5%)	3.5 $\pm$ (10%)	1.6 $\pm$ (20%)	<0.3
65	AAS	3.178 $\pm$ 0.194	40.91 $\pm$ 7.35	2.34 $\pm$ 0.164	-	0.34 $\pm$ 0.104
66	AAS	4.5 $\pm$ 0.4	34.5 $\pm$ 2	-	-	-
67	AAS	-	-	-	-	-
68	AAS	0.50	29.4	-	-	-
69	AAS	1.1	4.0	-	-	-
70	AAS	4.6 $\pm$ 1.24	25.1 $\pm$ 1.0	-	-	-
71	AAS	9.6	27.8	-	-	-
72	AAS	3.9 $\pm$ 0.3	31 $\pm$ 3	1.1	-	0.093 $\pm$ 0.01
73	AAS	-	-	-	-	-
	NAA	-	-	-	-	-
74	DFP	3.2	192.00	-	-	-
	ASV	4.68 $\pm$ 0.36	41.1 $\pm$ 1.7	-	-	-
75	AAS	4.09 $\pm$ 0.33	35.73 $\pm$ 0.24	-	-	-
76	AAS	3.69	35.6	-	-	-
77	AAS	3.42 $\pm$ 0.21	27.58 $\pm$ 1.10	1.94 $\pm$ 0.10	-	-
				2.05 $\pm$ 0.05		

TABLE 3 RESULTS OF TRACE ELEMENT MEASUREMENTS ON THE FISH FLESH SAMPLE MA-A-2
(Cd, Sb, Hg, Pb $\mu\text{g/g}$ -dry matter)
(SUPPLEMENT)

Code No.	Method	Cd	Sb	Hg	Pb
57	AAS	-	-	0.2229	-
58	AAS	0.34 $\pm$ 0.02	-	0.80 $\pm$ 0.06	-
	NAA	-	-	-	-
59	AAS	0.05 $\pm$ 0.01	-	0.49	< 0.45
60	NAA	-	-	-	-
61	AAS	< 0.24	-	-	< 5.5
62	NAA	0.07 $\pm$ 0.02	0.006 $\pm$ 0.002	0.49 $\pm$ 0.04	-
63	AAS	-	-	0.40 $\pm$ 0.05	-
64	NAA	< 2	< 0.005	3.5 $\pm$ (10%)	-
65	AAS	0.065 $\pm$ 0.017	-	0.393 $\pm$ 0.054	0.460 $\pm$ 0.045
66	AAS	< 0.3	-	0.58 $\pm$ 0.06	< 6
67	AAS	0.059 $\pm$ 0.008	-	0.523 $\pm$ 0.04	-
68	AAS	0.25	-	0.42	0.56
69	AAS	-	-	-	-
70	AAS	-	-	0.28 $\pm$ 0.05	-
71	AAS	1.1	-	0.474	5.1
72	AAS	0.061 $\pm$ 0.005	0.53 $\pm$ 0.06	1.1	0.58 $\pm$ 0.06
73	AAS	0.067	-	0.568	0.564
	NAA	0.055 $\pm$ 0.005	-	0.443 $\pm$ 0.016	-
74	DPP	< DL	-	-	0.94
	ASV	0.048 $\pm$ 0.005	-	-	1.44 $\pm$ 0.24
75	AAS	-	-	-	-
76	AAS	0.28	-	-	1.38
77	AAS	0.047 $\pm$ 0.006	-	-	0.22 $\pm$ 0.03

TABLE 4 OVERALL AVERAGES AND AVERAGES AFTER CHAUVENET AND DIXON'S TESTS
FOR THE RESULTS REPORTED FOR Cr, Mn, Fe, Co AND Ni MEASUREMENTS
ON FISH FLESH SAMPLE MA-A-2

Element	Cr	Mn	Fe	Co	Ni
No. of reported results*	36	41	52	25	23
Range reported ($\mu\text{g/g-dry}$)	0.259-12.9	0.44-3.5	18.6-158.15	0.03-2.3	0.42-2.54
Overall Average** ($\mu\text{g/g-dry}$)	2.5 ± 0.4 (16%)	1.0 ± 0.1 (10%)	57 ± 3 (5.3%)	0.5 ± 0.2 (40%)	1.1 ± 0.2 (18%)
<u>Chauvenet's Test</u>					
No. of results accepted by the test	26	36	47	18	23
Average** ($\mu\text{g/g-dry}$)	1.3 ± 0.1 (7.7%)	0.81 ± 0.04 (4.9%)	54 ± 1 (1.9%)	0.08 ± 0.01 (13%)	1.1 ± 0.2 (18%)
<u>Dixon's Test</u>					
No. of results accepted by the test	34	39	51	25	23
Average** ($\mu\text{g/g-dry}$)	2.0 ± 0.3 (15%)	0.88 ± 0.06 (6.8%)	55 ± 2 (3.6%)	0.5 ± 0.2 (40%)	1.1 ± 0.2 (18%)

* Results showing only detection limits of methods used are not included.

** The standard deviations given are 1 σ for mean (standard error).

TABLE 5 OVERALL AVERAGES AND AVERAGES AFTER CHAUVENET AND DIXON'S TESTS
FOR THE RESULTS REPORTED FOR Cu, Zn, As, Se AND Ag MEASUREMENTS
ON FISH FLESH SAMPLE MA-A-2

Element	Cu	Zn	As	Se	Ag
No. of reported results*	64	74	21	17	11
Range reported ($\mu\text{g/g-dry}$)	0.50-17.6	4.0-204	0.75-7.3	0.5-11.3	0.08-30
Overall Average** ($\mu\text{g/g-dry}$)	4.7 ± 0.3 (6.4%)	37 ± 3 (8.1%)	2.7 ± 0.6 (22%)	2.3 ± 0.6 (26%)	3 ± 3 (100%)
<u>Chauvenet's Test</u>					
No. of results accepted by the test	50	66	18	16	7
Average** ($\mu\text{g/g-dry}$)	4.0 ± 0.1 (2.5%)	33 ± 1 (3.0%)	2.6 ± 0.1 (3.8%)	1.7 ± 0.3 (18%)	0.10 ± 0.01 (10%)
<u>Dixon's Test</u>					
No. of results accepted by the test	63	72	20	16	10
Average** ($\mu\text{g/g-dry}$)	4.5 ± 0.3 (6.7%)	33 ± 1 (3.0%)	2.5 ± 0.2 (8.0%)	1.7 ± 0.3 (18%)	0.17 ± 0.04 (24%)

* Results showing only detection limits of methods used are not included.

** The standard deviations given are for mean (standard error).

TABLE 6 OVERALL AVERAGES AND AVERAGES AFTER CHAUVENET AND DIXON'S TESTS
FOR THE RESULTS REPORTED FOR Cd, Sb, Hg AND Pb MEASUREMENTS ON
FISH FLESH SAMPLE HA-A-2

Element	Cd	Sb	Hg	Pb
No. of reported results*	41	8	53	33
Range reported ($\mu\text{g/g-dry}$)	0.02-1.1	0.0032-0.53	0.179-3.5	0.097-5.1
Overall Average** ($\mu\text{g/g-dry}$)	0.17 ± 0.04 (24%)	0.09 ± 0.07 (78%)	0.54 ± 0.06 (11%)	0.9 ± 0.2 (22%)
<u>Chauvenet's Test</u>				
No. of results accepted by the test	29	5	51	29
Average** ($\mu\text{g/g-dry}$)	0.066 ± 0.004 (6.1%)	0.005 ± 0.001 (20%)	0.47 ± 0.02 (4.3%)	0.58 ± 0.07 (12%)
<u>Dixon's Test</u>				
No. of results accepted by the test	41	5	52	32
Average** ($\mu\text{g/g-dry}$)	0.17 ± 0.04 (24%)	0.005 ± 0.001 (20%)	0.49 ± 0.02 (4.1%)	0.8 ± 0.1 (13%)

* Results showing only detection limits of methods used are not included.

** The standard deviations given are 1 σ for mean (standard error).

ANNEX

LIST OF PARTICIPATING LABORATORIES
(SUPPLEMENT)

Laboratories which reported the results (29 February 1980)

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V. Blekastad

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G.L. Hoffman

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P.J. Whaling

Yugoslavia

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M. Branica

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J. Stirn
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PROGRESS REPORT No. 1
INTERCALIBRATION OF ORGANOCHLORINE COMPOUND
MEASUREMENTS IN MARINE ENVIRONMENTAL SAMPLES

International Laboratory of Marine Radioactivity

Musée océanographique
Principauté de Monaco

1. Participation in the Intercalibration Exercise

The occasion of this progress report is to inform all participants of the results which have been tabulated to date for the samples distributed. Because of the late arrival of data from a number of laboratories, the report has been somewhat delayed. It was judged more advisable, however, to try and include as many results as possible, rather than adhere to a rigid time schedule which would exclude valuable information. As originally proposed, the results contained in this report are listed by laboratory code number only, so that each participant can compare his analysis against the overall averages obtained.

At the outset of the programme fifty two laboratories were contacted. Six laboratories declined to analyze samples either because of previous commitments or because they felt that certain of the samples to be supplied could not be properly analyzed with the techniques which were in current use in their laboratories. Of the remaining forty-six institutions, eighteen have yet to reply. The twenty-eight institutes who did undertake organochlorine compound analyses did not analyze all samples which were available and therefore our original hope of having a large number of analyses to report has not been entirely realized. Listed below are the laboratories who have analyzed one or more samples, with the name of the principal investigator involved.

DENMARK

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Dr. M. Picer

2. Homogeneity tests for intercalibration samples

The Monaco Laboratory has prepared the following reference materials for intercalibration measurements:

<u>MATERIAL</u>	<u>COLLECTION SITE</u>
1. Oyster homogenate	Sète, France
2. Sea Plant homogenate	Mediterranean Coast
3. Sediment	Mediterranean Coast
4. XAD-2 resin	Mediterranean Coast
5. Plankton homogenate	Bergen, Norway
6. Fish Fillet homogenate	Atlantic Coast, France

Oyster (Crassostrea, sp.), Plankton (Calanus finmarchicus)

Fish fillet (Mugil cephalus), Sea Plant (Posidonia oceanica)

To date only the first four samples have been distributed to participating institutes. The plankton and fish fillet material are still undergoing homogeneity tests.

Following preparation of reference material, 1 through 4, homogeneity tests were conducted at the Monaco Laboratory by analyzing samples sizes that would yield sufficient amounts of organochlorine compounds for quantitation. In the case of the oyster sample, 10 samples weighing 10 grams each showed acceptable homogeneity for all compounds determined, i.e. a standard deviation of $\pm 20\%$ from the mean value obtained. The highest standard deviation was observed for pp'TDE (23%) while the lowest standard deviation was recorded for pp'DDT (2.8%). All other compounds gave standard deviations between these ranges and all were less than $\pm 15\%$. In addition, selected trace metals were determined in the sample to further test for homogeneity. Of eight trace metals analyzed, the largest standard deviation observed was 14% (Pb), while the smallest standard deviation noted was for Zn (2.1%). Other metals analyzed included Cd, Fe, Cr, Mn, Ni and Cu.

Similar homogeneity results were obtained for the sediment, sea plant and Amberlite samples. As a further precaution, however, the oyster and Amberlite XAD-II resin were tested for residue uniformity by a U.S laboratory

which confirmed the findings of the Monaco Laboratory. Repeated measurements of all samples over the past year have shown no change in pesticide residue or PCB content with time.

3. Preliminary survey of the results of the intercalibration exercise

Set out below are the results which have been received to date by laboratory code (TABLES 1-4). In addition, a statistical treatment of the data has been made to give overall ranges, mean and standard deviations for all compounds reported. Compared separately are the results of seven laboratories who have participated in previous intercalibration programmes (TABLES 10-13). This latter compilation is intended to show the comparability of the results of institutes who have been making organochlorine compound measurements on marine environmental samples for some time. We have chosen to tabulate all concentrations on a dry weight basis, rather than on an extractable organic matter basis, since there was a large variation in the amount of extractable organic matter reported for the biological materials. An example of this variation is shown in the results obtained for the oyster homogenate (TABLE 5).

Before treating the results statistically, it was necessary to make subjective decisions about data rejection. Despite an initial request for each participant to include information pertinent to the interpretation of the samples analyzed (including chromatograms for the analysis), many analysts did not comply with this request. Many results were reported as non-detectable or less than some given value, which had to be considered as a detection limit. Since the analysts did not include an explanation of these values, it was not possible to include them in the final statistical calculations, moreover, we have tabulated the PCB concentrations for all samples except the sediment in only those cases where the quantitation was reported as the 50% chlorine by weight mixture (Kanechlor 500, Aroclor 1254, Phenoclor DP-5 or Chlophen 50). Reference to the individual laboratory results shows that many analysts reported more than a single per cent by weight PCB mixture. In many cases, it was not possible to distinguish these percentages from the information supplied, and for this reason we have chosen to omit them from the statistical treatment. For similar reasons, the sediment values are tabulated for PCB concentrations which were reported as the 60% chlorine by weight mixture. The reader is therefore cautioned to view the results with the reservations as explained; compilation of the total PCB content of any sample analyzed would have yielded results more divergent than is suggested by the overall means and standard deviations reported in Tables 6-9.

4. Preparation of plankton and fish fillet samples

Within the next month, the plankton sample collected from Bergen, Norway, will be ready for dispatch to those institutes who wish to continue to participate in the intercalibration exercise. Within two months the fish fillet homogenate should be ready for distribution. Those participants who want to receive these samples are requested to contact the laboratory so that adequate preparation can be made for the packaging and mailing of the samples as soon as possible. We would also appreciate any comments relating to the presentation of the results in this progress report before their inclusion into the upcoming technical progress report of the laboratory. Please address all correspondence to:

Dr. D. Elder
International Laboratory of Marine Radioactivity
Musée océanographique
Principauté de Monaco

Table 1. Results of pesticide residue and PCB analyses for oyster tissue homogenate. MA-M-1 (ppb/dry weight).

Laboratory code	α BHC	β BHC	γ BHC	δ BHC	HCB	Dieldrin	Aldrin	Endrin	Heptachlore	Heptachlore epoxide
1	-	-	9	-	<1	20	-	-	-	-
4	-	-	-	-	-	20	-	70	-	-
6	2.8	-	9.4	-	-	4.7	-	-	-	-
7	<1	<5	7.5	-	<1	3	-	-	-	-
12	-	-	11	-	-	<10	-	-	-	-
13	0.15	-	26.9	-	-	26.9	-	-	-	-
15	2.1	2.6	10	-	2	20	-	-	-	6.1
16	-	11	7.3	-	-	-	-	-	-	-
18	-	-	13.5	-	-	<5	-	-	-	-
21	1	-	78	-	-	0.5	-	-	-	-
24	-	-	6.5	-	-	5.4	trace	-	trace	9.5
26a	-	-	-	-	-	8.5	-	-	-	-
26b	-	-	-	-	-	7.4	-	-	-	-
29	16.8	-	139.1	-	-	52	68.95	-	17.88	0.33
32	-	-	11	-	-	< 8	-	-	-	-
36	-	-	11.4	-	-	-	<0.6	-	-	-
43	66	-	35.27	8.9	-	-	9.2	-	-	-
44	7.5	-	13.1	2.3	-	5.2	-	-	-	-
45	2.2	-	12	-	-	-	-	-	-	-
46	-	-	3	-	-	15	-	-	-	-
47	2.7	-	4.3	-	-	19.1	-	-	-	-
49	-	-	20	-	-	-	-	-	-	-
50	-	-	0.93	-	-	-	-	-	-	-
52	1.8	-	9.5	-	-	-	-	-	-	-

14,17,20
23,25,37 NO RESULTS FURNISHED FOR ANY RESIDUES LISTED

- signifies that the laboratory reported no value for the pesticide residue or PCBs

Table 1. (cont'd.) (ppb/dry weight).

Laboratory code	pp'DDT	op'DDT	pp'TDE	pp'DDE	PCB	PCB characterization
1	71	-	31	79	426	aroclor 1254
4	50	-	-	200	2000	clophen A-30
6	63.7	-	29	40	340	aroclor 1242:1254 (1:1 mixture)
7	11	2	39	74.5	485	aroclor 1254
12	86	31	43	74	470	aroclor 1254
13	20.9	-	-	-	<300	aroclor 1254
15	79	-	47	87	260	clophen A-60
16	-	-	18	65	280	phenoclor DP-5
17	28	-	20	53	144	aroclor 1254
18	113.6	48.2	80.5	87	-	-
21	72	-	24	730	220	aroclor 1254
24	150	13	50	37	170	aroclor 1254
25	122	-	49	91	561	phenoclor DP-5
26a	105.2	-	46.8	87.6	523	aroclor 1242,48,54,60
26b	104	-	45.4	93.9	484	clophen A-60
29	-	141.35	-	30.23	-	-
32	109	-	45	76	470	aroclor 1254
36	-	-	132.9	61.1	370.9	aroclor 1254
37	44	-	-	150	190*+155**	aroclor 1254*, 1260**
43	160.8	107.2	-	192.6	434	kanechlor 600
44	-	-	29	64	450	kanechlor 300,500,600
45	48	14	29	98	420	kanechlor 500
46	22	12	9	30	86	kanechlor 500
47	120	-	-	95.44	510.9	aroclor 1248:1254 (1:1 mixture)
49	-	-	-	100	-	-
50	2.45	-	-	-	50	phenoclor DP-5
52	97	13.8	51	77	345	phenoclor DP-5
14,20,23	NO RESULTS FURNISHED FOR ANY RESIDUES OR PCBs LISTED					

Table 2. Results of pesticide residue and PCB analyses for sea plant homogenate SP-M-1. (ppb/dry weight).

Laboratory code	α BHC	β BHC	γ BHC	Dieldrin	Others
1	-	-	21	6	HCB: <1
4	-	-	-	10	Endrin: <10
6	0.5	-	7.7	-	-
7	< 2	<10	20	7	HCB: <1
12	-	-	14	-	-
13	0.14	-	22.57	533	-
15	0.92	1	12	10	HCB: 3.4/Heptoxide: 10
16	-	4.7	9.7	-	-
18	-	-	20	5.64	Heptoxide: 6.96
21	1	-	22	-	-
24	4.4	-	12	9	Heptachlore: 5.5/Aldrin: 6
26a	-	-	-	16.9	-
26b	-	-	-	16.7	-
29	42.15	-	8853.7	-	Aldrin: 566.3
32	<0.2	-	19	7	-
36	-	-	21.6	-	Aldrin: 220.9
43	56.11	-	90.64	87.7	-
44	10.2	-	19	8.6	δ BHC: 2.2/Aldrin: 2
45	0.61	-	9.9	-	-
46	-	-	18	14	-
47	3.72	6.04	13.6	33.4	Endrin: 8.98
49	-	-	20	-	-
50	-	-	0.27	-	-
52	-	-	20	-	-
20,23,25,37	NO RESULTS FURNISHED FOR ANY RESIDUES LISTED				

- signifies that the laboratory reported no values for the pesticide residues listed for PCBs.

Table 2. (cont'd.) (ppb/dry weight).

Laboratory code	pp'DDT	op'DDT	pp'TDE	pp'DDE	PCB	PCB characterization
1	23	-	7	12	202	aroclor 1254
4	10	-	-	-	500	clophen A-30
6	17.3	-	6.3	2	500	aroclor 1242:1254
7	6.5	<1	6.5	<10	215	aroclor 1254
12	21	-	-	<10	270	aroclor 1254
13	22.25	3.9	-	-	<110	aroclor 1260
15	14	-	5.4	7.4	85	clophen A-60
16	39	-	-	7	320	phenoclor DP-5
17	3	-	2	3	53	aroclor 1254
18	34.85	11.4	16.88	6.7	-	-
21	2	-	8	6	110	aroclor 1254
24	20	2	5	7	180	aroclor 1254
25	18	-	-	8	460 [*] +187 ^{**}	phenoclor DP-4 [*] , DP-5 [*]
26a	33.6	-	9.5	9.6	611	mixture
26b	44.2	-	14.5	11	295 [*] +681 ^{**}	clophen A-60 [*] , aroclor 1248 ^{**}
29	-	4934.10	-	-	-	-
32	23	-	6	3	150	aroclor 1254
36	-	-	120.6	8.8	1867.6	aroclor 1254
37	20	-	-	12	320 [*] +280 ^{**}	aroclor 1242 [*] /1254 ^{**}
43	43.8	-	-	-	470.2	kanechlor 500
44	21	12	-	40	450	
45	6.5	1.8	1.5	4.3	320	kanechlor 300,400,500 (1:1:1) mixture
46	-	-	-	7	120	kanechlor 500
47	103.6	-	-	16.1	548.7	aroclor 1248 and 1254 (1:1 mixture)
50	0.49	-	-	-	8.3	phenoclor DP-5
52	21	6.5	7	7	328 [*] +119 [*]	phenoclor DP-4 [*] /DP-5 [*]
14,20,23,49	NO RESULTS FURNISHED FOR ANY RESIDUES OR PCB'S LISTED					

Table 3. Results of pesticide residue and PCB analyses for sediment sample, SD-M-1. (ppb/dry weight).

Laboratory Code	α BHC	β BHC	γ BHC	Dieldrin	Others
4	-	-	-	20	Endrin; 50
6	0.1	-	0.7	1.2	
7	<10	<50	<10	1	HCB: <10
16	-	6.3	2.1	-	-
21	<0.5	-	1	-	-
26a	-	-	-	2.6	-
26b	-	-	-	2.5	-
29	348	-	314	15.2	Heptachlore: 337.8/Aldrin: 34.2 Heptachlore epoxide: 19
32	<0.3	-	<0.3	4	-
36	181	-	-	-	Aldrin: 1348
43	5.2	-	57.3	31.5	Aldrin: 8.6
44	2	-	1.2	2.5	δ BHC: 0.6
45	0.38	-	0.75	-	-
46	-	-	-	41	-
47	-	-	-	3.4	Endrin: 3.1

1, 12, 13, 14, 15,

17, 18, 20, 23, 24,

25, 37, 49, 50, 52

NO RESULTS FURNISHED FOR ANY RESIDUES LISTED.

- signifies that the laboratory reported no values for the pesticide residues listed, or for PCB's.

Table 3. (cont'd.) (ppb/dry weight)

Laboratory code	pp'DDT	op'DDT	pp'TDE	pp'DDE	PCB	PCB characterization
4	-	-	-	20	300	clophen A-30
6	7	-	3	2.6	237	aroclor 1242/1250
7	13	<1	11	<3	116.6*+156.6**	aroclor 1254*/1260**
13	10	-	-	-	<202	aroclor 1260
14	60	-	120	70	-	-
16	185	-	8.5	-	230	phenoclor DP-5
21	14	-	6	4	200	aroclor 1260
25	2	-	7	0.5	64*+238**	phenoclor DP4*/DP6**
26a	20.3	-	10.5	3.3	344	mixture
26b	17.5	-	10.9	2.4	255	clophen A-60
29	-	635	-	-	-	-
32	13	-	7	2	220	aroclor 1260
36	-	-	12	-	104	phenoclor DP-6
37	20	-	-	3.5	60*, 120**, 150***	aroclor 1242*, 1254** 1260***
43	-	-	-	-	335	kanechlor 600
44	25	14	10	-	300	kanechlor 300,500,600 (mixture 1:4:4)
45	15	1.4	8.5	4.7	270	kanechlor 600
46	10	-	8	3	133	kanechlor 500,600 (1:1)
47	30.9	-	-	5.34	270.3	aroclor 1248/1254 (1:1)
52	7.3	-	6.3	1.6	146	phenoclor DP-6

12, 15, 17, 18

20, 23, 24, 49,

NO RESULTS FURNISHED FOR ANY RESIDUES OR PCBs LISTED

50

Table 4. Results of pesticide residue and PCB analyses for Arberlite XAD-II resin. (ppb/dry weight).

Laboratory code	α BHC	β BHC	γ BHC	Dieldrin	Others
4	-	-	9.3	7.3	-
6	5	-	3.1	0.5	-
7	2	0.2	0.9	0.5	-
13	3	-	14	12.5	-
16	trace	57	7.6	-	-
21	7	-	-	-	-
23	10	-	2.5	-	-
29	4	-	229	-	Aldrin: 26.1
32	3.7	-	2.9	1.4	-
36	5.1	-	4.8	-	Heptoxide: 1.2/Aldrin: 5.8
49	20	-	20	-	-
50	6.6	-	5.8	-	-

1,12,14,15,
17,18,20,24,
25,26,37,43,
44,45,46,47

NO RESULTS FURNISHED FOR ANY RESIDUE LISTED

Table 4. (cont'd.) (ppb/dry weight)

Laboratory code	pp'DDT	op'DDT	pp'TDE	pp'DDE	PCB	PCB characterization
4	-	-	-	6.9	-	-
6	8.8	-	-	3.1	126	aroclor 1242/1250
7	<1	<1	<1	<0.5	21	aroclor 1254
13	14.4	-	-	-	-	-
14	270	-	-	-	-	-
17	<1	-	<1	<1	11	aroclor 1254
21	-	-	<1	<1	-	-
23	-	-	-	-	80	decachloro byphenyl
26a	-	-	-	0.3	57	mixture
26b	-	-	-	-	35.2	clophen A-60
29	-	83.5	-	-	-	-
32	0.5	-	0.2	0.6	17.3	aroclor 1254
36	-	-	-	-	78.3	aroclor 1254
37	-	-	-	-	47	aroclor 1254
50	-	-	-	-	1615	phenoclor DP-5
52	-	-	-	-	43.3	phenoclor DP-5

1,12,15,16,
18,20,23,24,
25,43,44,45,
46,47,49

NO RESULTS FURNISHED FOR ANY RESIDUE LISTED.

Table 5. Results on extractable organic matter and the ratio of PCBs to these values as reported by the various laboratories for oyster homogenate.

	Organic matter extractable concentration (mg/g)	(PCB) / (O.M) $\times 10^6$
Laboratories participating	26	26
N° of reported results	16	10
Maximum value	60	465.3
Minimum value	1.01	3.24
Overall average	32	59
σ	± 16	± 140
%	(50%)	(240%)

Table 6. Organochlorine compound concentrations in oyster tissue homogenate (MA-M-1). Reported values in ng/g (ppb) dry weight. (Laboratories reporting < values have been excluded.)

	α BHC	γ BHC	Dieldrin	pp'PPT	op'DDT	pp'DDD	pp'DDE	PCB (DP-5)
Laboratories participating	26	26	26	26	26	26	26	26
N° of reported results	10	21	14	22	8	19	24	14
Maximum value	66	139.1	52	160.8	141.35	132.9	730	561
Minimum value	0.15	0.93	0.5	2.45	12	9	30	50
Overall average	10	22	15	76	48	43	110	310
σ*	±20	±32	±13	±45	±50	±27	±140	±170
%	(200%)	(140%)	(87%)	(59%)	(100%)	(63%)	(130%)	(55%)
N° of results excluded by Dixon criteria	3	4	1	0	0	2	1	0
Range	0.15- 2.8	0.93- 20	0.5- 26.9	2.45- 160.8	12- 141.35	9-51	30-200	50-561
Overall average	1.8	9.4	12	76	48	36	88	310
σ*	±1	±4	±8	±45	±50	±13	±42	±170
%	(56%)	(43%)	(67%)	(59%)	(100%)	(36%)	(48%)	(55%)
N° of results excluded by Chauvenet criteria	3	6	1	0	4	2	4	0
Range	0.15- 2.8	3- 13.5	0.5- 26.9	2.45- 160.8	12-14	9-51	30-100	50-561
Overall average	1.8	9	12	76	13	36	74	310
σ*	±1	±3	±8	±45	±0.9	±13	±21	±170
%	(56%)	(33%)	(67%)	(59%)	(77%)	(36%)	(28%)	(55%)

*Standard deviation:

$$\sigma = \sqrt{\frac{\sum (X_i - \bar{X})^2}{N - 1}}$$

This equation is applicable to Tables 1 - 10.

Table 7. Organochlorine compound concentrations in sea plant (SP-M-1).
Reported values in ng/g (ppb) dry weight.
(Laboratories reporting < values have been excluded).

	α BHC	γ BHC	Dieldrin	Aldrin	pp'DDT	op'DDT	pp'DDD	pp'DDE	PCB(DP-5)
Laboratories participating	26	26	26	26	26	26	26	26	26
N° of reported results	10	21	14	4	23	7	14	19	15
Maximum value	56.11	8853.7	533	566.3	103.6	4934.1	120.6	40	1867.6
Minimum value	0.14	0.27	5.64	2	0.49	1.8	1.5	2	8.3
Overall average	12	440	55	200	24	710	15	9.4	300
σ	± 20	± 1900	± 140	± 270	± 21	± 1800	± 31	± 8.2	± 450
%	(170%)	(430%)	(250%)	(140%)	(88%)	(250%)	(210%)	(87%)	(150%)
N° of results excluded by Dixon criteria	0	2	3	0	1	1	1	2	1
Range	0.14- 56.11	0.27- 22.57	5.64- 16.9	2- 566.3	0.49- 44.2	1.8- 12	1.5- 16.88	2- 12	8.3- 470.2
Overall average	12	16	10	200	20	6.3	7.3	7.2	190
σ	± 20	± 6	± 4	± 270	± 13	± 4.5	± 4.3	± 3	± 120
%	(170%)	(38%)	(40%)	(140%)	(65%)	(71%)	(59%)	(42%)	(63%)
N° of results excluded by Chauvenet criteria	3	3	3	0	1	1	3	2	2
Range	0.14- 4.4	7.7- 22.57	5.64- 16.9	2- 566.3	0.49- 44.2	1.8- 12	1.5- 9.5	2- 12	8.3- 320
Overall average	1.6	17	10	200	20	6.3	5.8	7.2	170
σ	± 1.7	± 4.8	± 4	± 270	± 13	± 4.5	± 2.4	± 3	± 90
%	(110%)	(28%)	(40%)	(140%)	(65%)	(71%)	(41%)	(42%)	(53%)

Table 8. Organochlorine compound concentrations in Mediterranean Sediment (SD-M-1). Reported values in ng/g (ppb) dry weight. (Laboratories reporting < values have been excluded).

	α BHC	γ BHC	Dieldrin	pp'DDT	pp'DDD	pp'DDE	PCB(DP-6)
Laboratories participating	20	20	20	20	20	20	20
N° of reported results	6	7	11	16	14	13	10
Maximum value	348	314	41	185	120	70	335
Minimum value	0.1	0.7	1	2	3	0.5	104
Overall average	89	54	11	28	16	9.5	210
σ	± 150	± 120	± 14	± 44	± 30	± 19	± 70
%	(170%)	(220%)	(130%)	(160%)	(190%)	(200%)	(33%)
N° of results excluded by Dixon criteria	0	3	0	2	1	2	0
Range	0.1-348	0.7-1.2	1-41	2-30.9	3-12	0.5-5.34	104-335
Overall average	89	0.9	11	15	8.4	3	210
σ	± 150	± 0.2	± 14	± 7.7	± 2.5	± 1.4	± 70
%	(170%)	(22%)	(130%)	(51%)	(30%)	(47%)	(33%)
N° of results excluded by Chauvenet criteria	2	3	4	3	2	2	0
Range	0.1-5.2	0.7-1.2	1-4	2-25	6-12	0.5-5.34	104-335
Overall average	1.9	0.9	2.5	13	8.8	3	210
σ	± 2.3	± 0.2	± 1.1	± 6.3	± 2	± 1.4	± 70
%	(120%)	(22%)	(44%)	(48%)	(23%)	(47%)	(33%)

Table 9. Organochlorine compound concentrations in Amberlite XAD-II resin (AB-M-1), concentrations in ng/g (ppb) dry resin.
(Laboratories reporting < values have been excluded).

	α BHC	γ BHC	Dieldrin	pp'DDT	pp'DDE	PCB (DP-5)
Laboratories participating	19	19	19	19	19	19
N° of results reported	10	12	5	4	4	7
Maximum value	20	229	12.5	270	6.9	1615
Minimum value	2	0.9	0.5	0.5	0.3	11
Overall average	6.6	26	4.4	73	2.7	260
σ	± 5.2	± 64	± 5.3	± 130	± 3.1	± 600
%	(79%)	(250%)	(120%)	(180%)	(110%)	(230%)
N° of results excluded by Dixon criteria	1	2	0	1	0	1
Range	2-10	0.9-1.4	0.5-12.5	0.5-14.4	0.3-6.9	11-78.3
Average	5.2	5.8	4.4	7.9	2.7	36
σ	± 2.4	± 3.9	± 5.3	± 7	± 3.1	± 25
%	(46%)	(67%)	(120%)	(89%)	(110%)	(69%)
N° of results excluded by Chauvenet criteria	2	3	1	0	0	2
Range	2-7	0.9-9.3	0.5-7.3	0.5-270	0.3-6.9	11-47
Average	4.6	4.9	2.4	73	2.7	28
σ	± 1.7	± 2.6	± 3.3	± 130	± 3.1	± 16
%+	(37%)	(53%)	(140%)	(180%)	(110%)	(57%)

Table 10. Organochlorine compound concentrations in oyster homogenate (MA-M-1) as measured by seven selected laboratories who participated in previous intercalibration exercises. Reported values in ng/g (ppb) dry weight. (Laboratories reporting < values have been excluded).

	α BHC	γ BHC	Dieldrin	pp'DDT	op'DDT	pp'DDD	pp'DDE	PCB (DP-5)
N° of reported results	2	6	4	7	1	6	7	5
Maximum value	2.8	11	20	109	-	47	150	485
Minimum value	2.1	7.5	3	11	-	29	40	190
Overall average	2.5	9.7	12	66	31	39	83	410
σ	± 0.5	± 1.3	± 9.4	± 32	-	± 7.5	± 33	± 120
%	(20%)	(13%)	(78%)	(48%)	-	(19%)	(40%)	(29%)

Table 11. Organochlorine compounds concentrations in Sea Plant (SP-M-1) as measured by seven selected laboratories who participated in previous intercalibration exercises. Reported values in ng/g (ppb) dry weight. (Laboratories reporting < values have been excluded).

	α BHC	γ BHC	Dieldrin	Aldrin	pp'DDT	op'DDT	pp'DDD	pp'DDE	PCB (DP-5)
N° of reported results	2	6	4	0	7	0	5	5	5
Maximum value	0.92	20	10	-	23	-	7	12	280
Minimum value	0.5	7.7	6	-	6.5	-	5.4	2	150
Overall average	0.71	16	7.5	-	18	-	6.2	7.3	220
σ	± 0.29	± 5.2	± 1.7	-	± 5.9	-	± 0.6	± 4.7	± 53
%	(41%)	(33%)	(23%)	-	(33%)	-	(9.7%)	(64%)	(24%)

Table 12. Organochlorine compound concentrations in sediment sample (SD-M-1) as measured by seven selected laboratories who participated in previous intercalibration exercises. Reported values in ng/g (ppb) dry weight. (Laboratories reporting < values have been excluded).

	α BHC	γ BHC	Dieldrin	pp'DDT	pp'DDD	pp'DDE	PCB(DP-6)
N° of reported results	1	1	3	4	3	3	3
Maximum value	-	-	4	20	11	3.5	220
Minimum value	-	-	1	7	3	2	150
Overall average	0.1	0.7	2.1	13	7	2.7	180
σ	-	-	± 1.7	± 5.3	± 4	± 0.7	± 39
%	-	-	(81%)	(41%)	(57%)	(26%)	(22%)

Table 13. Organochlorine compound concentrations in Amberlite XAD-II resin (AB-M-1) as measured by seven selected laboratories who participated in previous intercalibration exercises. Reported values in ng/g (ppb) dry weight resin. (Laboratories reporting < values have been excluded).

	α BHC	γ BHC	Dieldrin	pp'DDT	pp'DDE	PCB (DP-5)
N° of reported results	3	3	3	2	2	3
Maximum value	5	3.1	1.4	8.8	3.1	47
Minimum value	2	0.9	0.5	0.5	0.6	17.3
Overall average	3.6	2.3	0.8	4.7	1.9	28
σ	± 1.5	± 1.2	± 0.5	± 5.9	± 1.8	± 16
%	(42%)	(52%)	(63%)	(130%)	(95%)	(57%)

Lab. N°	Method of Extraction			Clean up			Pre G.L.C. separation	Gas Liquid Chromatography (G.L.C.)			Confirm. Chemical Test
	Cold	Hot	Time	Partition	Column	Other		Packing	Length	Temperat.	
1		Hexane	4 hrs		Alumina		a) hexane b) E.E/hexane (1:9)	5% DCFS 1265 + 4% DC 200 Chromosorb w 80/100 10% DC 200 chromosorb w 80/100			
4		P.E.	5 hrs					3% ov 210 + 3% ov 17 Gas chrom. Q 100/120 PCB: 10% DC 200 15% QF1 Chromosorb w 80/100	2	185	
6		SP/1E	4 hrs		Florisil		a) P.E. b) P.E/S.E (80:20)	10% DC 200 chromosorb w 80/100 6% QF1 chromosorb w 80/100 10% ov 17 10% ov 210 (1:4) chromosorb w 80/100	1.5 3 3		
7		Hexane/ Acetone (6:40)	2 hrs		Alumina			6% DC 200 or 4% DC 200 + 6% QF1 chromosorb w 80/100	2	200	12
12		E.E/P.E (1:90)	5 hrs		Florisil			2% ov 101 Gas chrom. Q 100/120 0.75% ov 17 + 0.97% ov 210 gas chrom. Q	1.82	130	+
13		P.E.			Florisil	Alcohol- lic KOH CR ₂ O ₃	6% and 15% E.E in P.E.	7% QF1 + 5% DC 200 chromosorb w 1.5% ov 17 + 1.95% QF1 chromosorb w 80/100			
14		E.E/Hexane (15:85)	72 hrs		Florisil			5% QF1 varaport 30 80/100 2.5% QF1 + 2.5% DC 200 varaport 30 60/100	2	200	
15	P.E. CH ₂ CL ₂ (9:2) in column							S.E 30	60		
16	P.E.				Florisil						

Lab. N°	Method of Extraction			Clean up			Pre G.L.C. separation	Gas Liquid Chromatography (G.L.C.)			Confirm Chemical Test
	Cold	Hot	Time	Partition	Column	Other		Packing	Length	Temperat.	
17	Blending P.E.				Alumina		Pentane Benzene on silica gel	1.5% SP 2250 + 1.95% SP 2401 supelcon aw 4% SE 30 + 6% ov 210 gas chrom. Q	1.8 1.5	210	
18		P.E.	5 hrs		Florisil	P.E./ACN (1:2)	6% and 15% E.E. in P.E. + celite sea sorb column	3% DC 200 + 5% QF1 (1:1) gas chrom. Q	1.8		
20					Florisil			2% DC 200 + 2.5 QF1 varaport 30	metal 1.8	185-200	
21		Acetone/hexane (60:40)	12 hrs		Silver Nitrate Alumina column			2% ov 1 + 3% QF1 chromosorb w 80/100	1.5	200	129
23								5% SE 30 varaport 30	0.6 inox	210	
24		P.E./acetone (1:1)	8 hrs	ACN/P.E.	Florisil			1.5% ov 17 + 1.95% QF1 gas chrom. Q 100/120	1.8	200	+
25		Hexane	9 hrs		Florisil	H ₂ SO ₄ Hg for sediment		3% ov 1 Gas chrom. Q 80/100		180	+
26	Hex. and acetone in column		2 hrs		Florisil			5% SE 30 chromosorb w 80/100 + 5% SP 2250 + 1.95% SP 2401 supelcoport 100/120	2 1.7	220 190	
29	Blending water ACN (35:65)			P.E.	Florisil		6% and 15% E.E. in P.E.	1+ ov 17	1.7	185	

Lab. N°	Method of Extraction			Clean up			Pre G.L.C. separation	Gas Liquid Chromatography (C.L.C.)			Confirm. Chemical Test
	Cold	Hot	Time	Partition	Column	Other		Packing	Length	Temperat.	
32		Hexane	2 hrs		Florisil			4% DC 200 + 6% QF1 chrom. w 80/100 4% DC 200 chrom. w 80/100	1.8	180-185	
49	Ultra son P.E.				Florisil			3% ov 1 Chromosorb w 80/100	2	200	
50		Hexane	10 hrs	Hexane- ACN	Florisil			10% ov 101 Varapor 30 80/100		210	

S.E. = Sulfuric ether

E.E. = Ethyl ether

P.E. = Petroleum ether

A.C.N. = Acetonitrile

Lab. N°	Method of Extraction			Clean up			Pre G.L.C. separation	Gas Liquid Chromatography (G.L.C.)			Confirm. Chemical Test
	Cold	Hot	Time	Partition	Column	Other		Packing	Length	Temperat.	
36		Hexane	6 hrs		Florisil		silica gel a) hexane b) hex./E.E. (90:10)	7.5% QF1 + 5% DC 200 chromosorb w 80/100	1.3	180	+
37		ANC	24 hrs	ACN/hex.	Silica- alumina	Alcohol- lic KOH	10% benzene in hexane 20% benzene in hexane	3% Apiezon L chromosorb w 80/100	2	230	+
43		Saponifi- cation	1 hr	Hexane	Silica gel		15% E.E. in P.E.	2% ov 1 Gas chrom. Q 80/100	2	180	
44								2% DC 200 Gas chrom. Q 1.4% ov 17 + 2.2% DC GF1 gas chrom. Q	1.5 2	200 205	- 131 -
45		E.E			Florisil	H ₂ SO ₄	a) hexane b) E.E./ hexane (30:70)	2% ov 1 chromosorb w 80/100	2	180	
46	Blending water ACN		5 mn	Hexane	Silica gel		a) hexane b) E.E/hex. (15.85)	2% DEGS 0.5% PA 2% ov 17		180 190	+
47	Blending ethanol- hexane (1:1)		5 mn		Florisil	Hexane alcohol- lic KOH ether (5:1)	Silica gel a) hexane b) E.E./hex. (3:17)	1.5% ov 17 Chromosorb w	1.5	180 190	

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PROGRESS REPORT ON INTERCALIBRATION OF ORGANOCHLORINE COMPOUND MEASUREMENTS
IN MARINE ENVIRONMENTAL SAMPLES

International Laboratory of Marine Radioactivity

Musée Océanographique

Principauté de Monaco

No. 2 - April 1980

Intercalibration Exercises on Copepod (MA-A-1) and Fish (MA-A-2) Samples

The Monaco Laboratory prepared the following intercalibration samples for chlorinated hydrocarbon measurements during the past few years:

- 1) oyster homogenate (MA-M-1)
- 2) sea plant homogenate (SP-M-1)
- 3) sediment (SD-M-1)
- 4) XAD-2 resin
- 5) copepod homogenate (MA-A-1)
- 6) fish fillet homogenate (MA-A-2)

The results of the intercalibration exercises on the samples (1) - (4) have already been compiled, based on the reports by 26 laboratories, and have been distributed to participating laboratories.

The second set of the samples, (5) and (6), the copepod sample (MA-A-1) (Calanus cristatus) collected from the Norwegian Sea and the fish fillet sample (MA-A-2) prepared from the material obtained from the French Atlantic coast, was sent to the laboratories who expressed their willingness to continue the intercalibration exercises. To date 15 laboratories have sent their results. A list of these laboratories is attached to this report. The results presented in this report are listed by laboratory code number only, in order to make it possible for each participant to compare his analysis against the overall averages obtained.

Many results were reported as non detectable or less than the detection limits. It was not possible to include them in the final statistical calculations.

One of the two samples analyzed here is at a relatively high concentration level (fish fillet). For this the comparability of measurements is, in general, much better than that for the other sample.

In the second sample (copepod) the concentration levels of organochlorine compounds are low and the comparability of some results is less satisfactory. (eg. Table 3, pp'DDE and pp'DDD).

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Table 1. Results of pesticide residue and PCB analyses for copepod homogenate MA-A-1 (ppb/dry weight).

Lab. Code No.	Aroclor 1254 1260		PP' DDT	PP' DDE	PP' DDD	Aldrin	Dieldrin	α BHC	γ BHC	HCB	Others
1	181	N.R.	13	6	104	N.R.	15	N.R.	N.R.	N.R.	
3	212	N.R.	N.D.	5,26	N.D.	N.D.	3,07	N.R.	7,46	N.R.	
6	250	N.R.	<50	60	<50	<10	<20	10	5	N.R.	Penitrothion 25
7	200	<40	19	11	<1	<1	18	3	18	1	op'DDT: 6 Hept.epox.: 4
13	N.R.	TR.	TR.	14	TR.	N.R.	N.R.	N.R.	17	N.R.	
17	153	N.R.	0,59	1,5	<0,1	N.R.	4,7	N.R.	N.R.	N.R.	
24	190	110	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	N.R.	
26(a)	50	50	N.R.	5	N.R.	N.R.	N.R.	21	21	0,8	
26(b)	270	150	25	14	20	2	14	N.R.	N.R.	N.R.	
32	<1000		<300	<10	<40	N.R.	N.R.	<50	<50	<4	
36	348	N.R.	16	7	25	32		N.R.	N.R.	N.R.	
45		*	19	10	11	N.D.	5,1	16	22	1,1	op'DDT: 8 op'DDD: 6
46		**	21	26	13	N.D.	21	N.R.	N.R.	N.R.	
50	N.R.	N.R.	23	34	N.R.	N.R.	N.R.	9	20	N.R.	
53	71	N.D.	4	3	N.D.	N.R.	N.R.	4,5	5,6	N.R.	

N.R.: no results

N.D.: not detected

- : not interested

* : mixture KC 300 400 500 600 (4:4:6:1) : 360

** : KC 500 : 185

TR. : trace

Table 2. Results of pesticide residue and PCB analyses for fish fillet homogenate MA-M-2 (ppb/dry weight).

ab.Code No.	Aroclor		pp' DDT	pp' DDE	pp' DDD	Aldrin	Dieldrin	α BHC	γ BHC	HCB	Others
	1254	1260									
1	7147	N.R.	565	240	300	N.R.	122	N.R.	N.R.	N.R.	
3	4626	N.R.	98,9	87,7	161,1	N.D.	57,4	N.R.	23,9	36,69	
6	3500	5000	900	200	<50	<10	<20	10	70	N.R.	
7	7840	3020	280	170	380	N.R.	160	25	38	18	op'DDT: 26 Hept.epox: 30
13	16300		73	739	898	N.R.	N.R.	N.R.	93	N.R.	
17	987	N.R.	391,5	308,5	451,5	N.R.	102,7	N.R.	N.R.	N.P.	
24	15030	8800	140	270	80	N.R.	N.R.	N.R.	N.R.	N.R.	
26(a)	1340	6260	330	290	670	N.R.	210	41	62	18	Hept.epox: 41
26(b)	N.R.	6730	330	390	450	4	170	N.R.	N.R.	N.R.	
32	<1000		<200	<10	<10	N.R.	N.P.	<50	<50	<4	
36	7657	N.R.	305	664	554	<2,5	N.R.	N.R.	N.R.	N.P.	
45	*		160	250	310	N.D.	190	41	42	19	
46	**		257	262	308	62	168	N.R.	N.R.	N.P.	op'DDT: 27 op'DDD: 20
50	N.R.	1720	N.R.	N.R.	N.R.	N.P.	N.R.	20	33	N.R.	
53	4350	6750	N.D.	220	46	N.R.	N.R.	N.D.	88	N.R.	

N.R.: no results

N.D.: not detected

* : mixture KC 300, 500, 600 (1:4:2) : 12000

** mixture KC 400, 500, 600 (1:1:1) : 13540

Table 3. Organochlorine compound concentrations in copepod homogenate MA-A-1.
Reported values in ng/g (ppb) dry weight.
Laboratories reporting < values have been excluded.

	Aroclor 1254	Aroclor 1260	pp'DDT	pp'DDE	pp'DDD	ECB	Dieldrin	α BHC	γ BHC
No. of labor. participating	15								
No. of results	10	3	9	13	5	3	7	6	8
Maximum value	348	150	25	60	104	1,1	21	21	22
Minimum value	50	50	0,59	1,5	11	0,8	3,07	3	5
Overall average	190	100	16	15	35	1	12	11	15
σ	± 89	± 50	± 8	± 16	± 39	$\pm 0,2$	± 7	± 7	± 7
%	(47%)	(50%)	(50%)	(110%)	(110%)	(20%)	(58%)	(64%)	(47%)
No. of results after Chauvenet's test	10	-	9	10	4	-	7	6	8
Chauvenet's range	50-348	-	0,59-25	1,5-14	11-25	-	3,07-21	3-21	5-22
Average	190	-	16	8	17	-	12	11	15
σ	± 89	-	± 8	± 4	± 6	-	± 7	± 7	± 7
%	(47%)	-	(50%)	(50%)	(35%)	-	(58%)	(64%)	(47%)
No. of results after Dixon's test	10	-	9	11	4	-	7	6	8
Dixon's range	50-348	-	0,59-25	1,5-26	11-25	-	3,07-21	3-21	5-22
Average	190	-	16	9	17	-	12	11	15
σ	± 89	-	± 8	± 7	± 6	-	± 7	± 7	± 7
%	(47%)	-	(50%)	(78%)	(35%)	-	(58%)	(64%)	(47%)

Table 4. Organochlorine compound concentrations in fish fillet homogenate MA-M-2.
Reported values in ng/g (ppb) dry weight.
Laboratories reporting < values have been excluded.

	Aroclor 1254	Aroclor 1260	pp'DDT	pp'DDE	pp'DDD	HCB	Dieldrin	α BHC	γ BHC
No. of laborat. participating	15								
No. of results	9	7	12	13	12	4	8	5	8
Maximum value	15030	8800	900	739	898	36,69	210	41	93
Minimum value	987	1720	73	87,7	46	18	57,4	10	23,9
Overall average	5830	5470	319	315	384	23	148	27	56
σ	± 4280	± 2420	± 229	± 187	± 245	± 9	± 50	± 14	± 26
%	(80%)	(44%)	(72%)	(59%)	(64%)	(39%)	(34%)	(52%)	(46%)
No. of results after Chauve- net's test	8	7	10	9	11	3	8	5	8
Chauvenet's range	987-7840	1720- 8800	73-391,5	170-308,5	46-670	18-19	57,4-210	10-41	23,9-93
Average	4680	5470	237	245	337	18	148	27	56
σ	± 2700	± 2420	± 110	± 44	± 192	$\pm 0,6$	± 50	± 14	± 26
%	(58%)	(44%)	(46%)	(18%)	(57%)	(3%)	(34%)	(52%)	(46%)
No. of results after Dixon's test	8	7	11	11	12	3	8	5	8
Dixon's range	987-7840	1720- 8800	73-565	87,7-390	46-898	18-19	57,4-210	10-27,4	23,9-93
Average	4680	5470	266	244	384	18	148	27	56
σ	± 2700	± 2420	± 144	± 78	± 245	$\pm 0,6$	± 50	± 14	± 26
%	(58%)	(44%)	(54%)	(32%)	(64%)	(3%)	(34%)	(52%)	(46%)

Table 5. Results of percentage water and percentage extractable organic matter in the samples.

Laboratory code number	Copepod MA-A-1		Fish fillet MA-M-2	
	% water	% E.O.M.	% water	% E.O.M.
1	5,41	21,46	3,84	16,10
3	N.R.	21,93	N.P.	15,95
6	3,2	21,8	2,92	17,54
7	5,7	20,9	2,90	15,6
13	N.R.	21,7	N.R.	19,2
17	15,6	16,7	9,3	12,3
24	3,35	21,78	1,85	15,5
26(a)	4,91	14,15	2,88	16,26
26(b)	4,91	14,15	2,88	16,26
32	N.E.	N.R.	N.P.	N.E.
36	5,57	23,47	5,03	16,88
45	4,0	22,4	4,2	15,5
46	5,88	24,5	6,24	17,0
50	2,1	N.R.	0,42	N.R.
53	6,9	1,99	4,5	1,63

Lab. code	Drying procedure	Extraction	Confirmation
1	6 hours at 105°C	soxhlet, hexane	2 columns: -5% QF1, 4% DC 200 -5% QF1, 4% SF 96
3	Homogenized with sodium sulfate	soxhlet, hexane, 4 hours	GC/MS
6	Taken as such	soxhlet 4 hours Petroleum ether, diethyl/ ether (1+1)	OV 101, OV 17 + OV 210, QF1, OV 1 third Florisil clean-up GC/MS fragmentometry
7	Overnight at 35°C, then 105°C to constant weight	soxhlet, hexane, 1 1/2 hour	alcoholic KOH hydrobromic acid/acetic anhydride
13	Taken as such	cold extraction	alcoholic KOH
17		blender, hexane	alcoholic KOH
24		soxhlet, petroleum ether, 8 hours	alcoholic KOH
26	1 hour at 100°C (separate sample)	cold column (sample + Na ₂ SO ₄ hexane/acetone (2:1)	2 GLC columns
32		soxhlet, hexane, 1-2 hours	GC/MS
36	8 hours at 110°C	soxhlet, hexane, 8 hours	alcoholic KOH
45	1 hour at 100°C	soxhlet, ethyl ether, sample + Na ₂ SO ₄	
46	4 hours at 100°C	soxhlet, hexane	2 columns: OV 1 DEGS-PA or OV 17
50	5 hours at 103°C	soxhlet, petroleum ether, 8 hours	alcoholic KOH
53	sample not dried for organochlorine extraction	soxhlet, petroleum ether, 8 hours	

Lab. code	Work-up procedure	Gas chromatographic conditions
1	Al_2O_3 (Method of Holden).	Detector: 210°C, oven: 200°C, injector: 230°C
3	Chromatography on alumina (5% water) with hexane. Chromatography on silica (3% water) hexane and 10% ether in hexane.	Detector: 200°C, 4% SE 30.
6	1) (Fat + 2) + 8g Florisil (5% H_2O). Elution 100 ml (acetone + H_2O) (80 + 20) Transfert acetone, water $\longrightarrow$ P.E. 2) 10g-florisil (5% H_2O) E ₁ : 100 ml petroleum ether E ₂ : 60 ml (P.E., 20% ether).	
7	Method of Holden using clean-up and separation on alumina and silica into 4 eluates.	Detector: 200°C. 10% DC 200 or 5% DC 200 + 7,5% OF1 on 80-100 chromosorb W (HP).
13	Concentrated H_2SO_4 .	Detector: 280°C, oven 230°C, injector 248°C. 1,5% OV 17 + 1,95% OV 210 on varaport 30 80-100.
17	Alumina (5% water). Elution with 15 ml hexane. Then silica gel separation by Snyder-Reinert procedure.	Detector: 250°C, injector: 220°C 4% SE 30 + 6% OV 210 on gas chrom. Q 1,5% SP 2250 + 1,95% SP 2401 on 100/120 supelcon AW - DC'S.
24	Concentrated H_2SO_4 .	Detector: 250°C, oven: 205°C, injector: 240°C. 10% DC 200 on gas chrom. Q 80-100.
26	Florisil 1) hexane 2) hexane/ether (1:1) Removal of fat with Al_2O_3 column.	Fraction 1) isotherm: 197°C Fraction 2) temp. progr. 150-197°C (6°C/min). 1,5% SP 2250 + 1,95% SP 2401 on supercoport 80-100.
32	Alumina column (method of Holden).	
36	Silica gel (5% water) hexane 15 ml hexane 35 ml. Concentrated H_2SO_4 .	Detector: 290°C, oven: 190°C 3% OV 1 on chromport XXX 80-90.
45	Florisil (removal of fat and oil) Silica gel 1) hexane 2) 30% ether in hexane. Fuming H_2SO_4 .	Detector: 230°C, oven: 185°C (PCB) 2% OV 1. Detector: 190°C, oven: 190°C (pesticides) 2% OF1 + 1,5% OV 17.
46	Dry florisil chromatography (30% $\text{H}_2\text{O}/\text{CH}_3\text{CN}$, 200 ml). Silica gel chromatography.	Oven: 180°C. 2% DEGS + 0,5% PA.
50	Florisil (3% water).	Oven: 210°C 5% OV 17. Oven: 215°C 5% OV 101.
53	Concentrated H_2SO_4 .	Detector: 350°C, oven 200°C; injector 220°C. 10% DC 200 on chromosorb W.