See Letter Study EPA-ERT Edison N.J. (9/82 (?)) New Bedford Site 51732

SUMMARY

One of the most difficult aspects of PCB quantitation of environmental samples is to obtain a match between the sample and the standard. Environmental samples seldom have a GC peak pattern that will match any Aroclor standard, and even commercial preparation of PCB's vary from batch to batch in the abundance of minor components. In addition detector responce to different PCB isomers can vary by as much as 10,000 fold so that accurate quantitation is impossible. The retention time window method is a partial solution but is also not without it's faults especially with samples that may contain slight interferences.

The mechanism the change a PCB mixture in the environment are many, such as biodegredation of lighter isomers, partitioning across membranes and into the surronding aquatic environment, chemical degradation and selective non-reversable adsorbtion into a matrix to name a few.

The PCB's in the sea lettuce tissue itself resemble to some degree an Aroclor 1248 standard. However a weathered Aroclor 1242 and Aroclor 1254 may produce a very similar fingerprint. The concentrations stated in table II.1 are only approximations since an accurate fingerprint match to the standard are not possible in this case. The approximation can be said to be accurate to well within an order of magnitude of concentration and may deviate only by a few percent when compared to other methods.

SDMS DocID 51732

Analysis Protocol for Sea Lettuce Samples from New Bedford, Mass

I. SURFACE EXTRACT

a. Evaluate the surface area of a 4-5 gram portion of sea lettuce by carefully laying on a reasonably circular piece of sea lettuce on a glass plate, minimizing the folding, and measure the radius in several places and determine a mean. The surface area may be computed by the equation:

$$\text{SURFACE AREA} = 2 \text{ pi } r^2$$

b. Transfer the sea lettuce sample to a 125 ml. Erlenmeyer flask and then wipe up the glass plate with a fibre-glass filter pad moistened with 0.5% sodium lauryl sulfate solution and add the filter pad to the flask.

c. Wash the sea lettuce with three 15 ml. portions of 0.5% sodium lauryl sulfate solution, each time transfering the sediment containing extract to a new 125 ml. Erlenmeyer flask.

d. Evaporate the water in a 100°C oven overnight.

e. Extract the residue with three 15 ml. portions of pure pesticide grade hexane and transfer the organic extract to a 300 ml. round bottom flask for concentration in a rotary evaporator.

f. Evaporate the sample to dryness over steam and under vacuum.

g. Reconstitute the residue with 1 ml. of hexane. With a 1 ml. disposable pipet transfer 0.6 mls. of the concentrate into a hexane washed florisil SEP-PAK cartridge (Waters Assoc.) and elute the cartridge with 4.4 mls. of hexane. The concentration of PCB in the sample may be determined by the following equation:

PCB sample = PCB extract x 0.6/5.0 x surface area sample

or

PCB sample = PCB extract x 0.6/5.0 x weight sample

II. PLANT EXTRACT

a. Determine the weight of the plant material by weighing the tared Erlenmeyer flask containing the sea lettuce sample.

b. Transfer the sea lettuce to a Waring blender containing approx. 10 grams of anhydrous sodium sulfate. Rinse the Erlenmeyer flask with three 15 ml. portions of hexane and add that to the blender. Blend sea lettuce with solvent for approx. 5 mins., and then transfer the extract to a 300 ml. round bottom through a hexane wet filter. Rinse the blender with a 15 ml. portion of solvent and rinse the sea lettuce-sodium sulfate slurry with it.

c. Follow steps f. & g. section I. for concentration by rotary evaporation and clean-up.

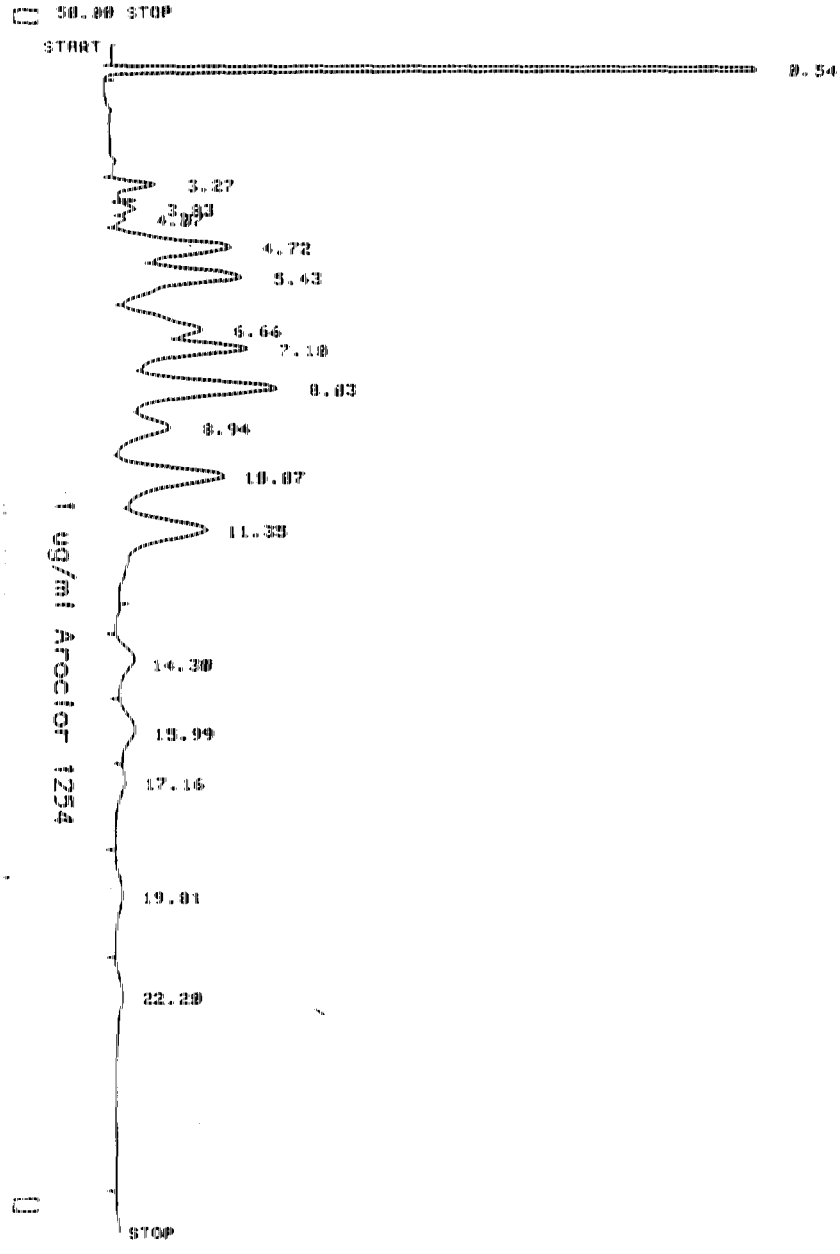
III. PCB ANALYSIS

a. Make a calibration range of Aroclor 1260 standards: 0.4 ug/ml. thru 2.0 ug/ml.

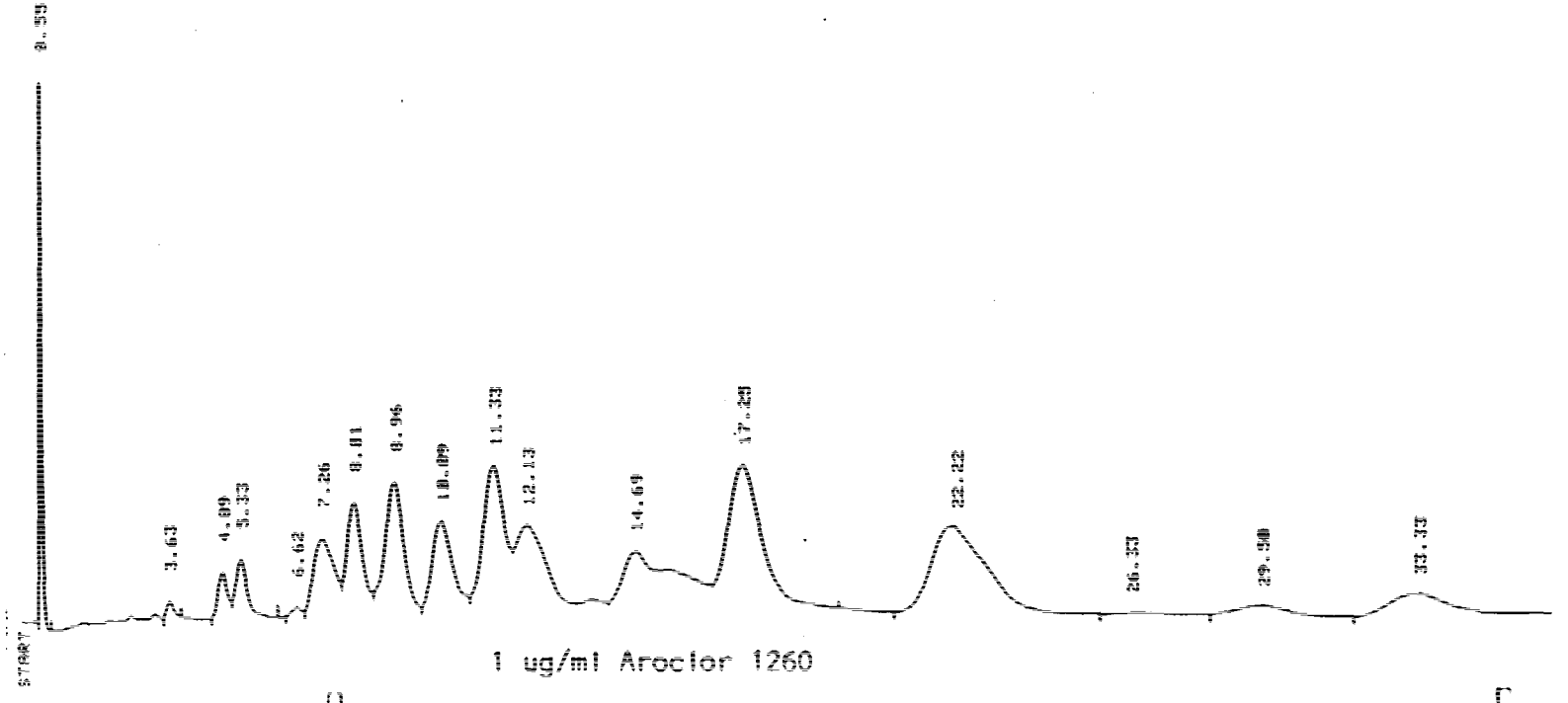
b. Make 1 ug/ml. stds. of Aroclor 1242, 1248, 1254, and 1260 to be run as fingerprinting standards and for quantitation.

c. Quantify by method of external standards using peak height for responce measurement.

d. The instrument used for chromatogrphy is an HP 5480 with auto sampler and ECD. The conditions used for analysis are:



HP RUN 0 107 OCT/01/92 TIME 03:40:04
ID:63091.2 BOTTLE 32
ESTD


9.99
3.63
4.89
5.33
6.62
7.26
8.01
8.96
10.09
11.33
12.13
14.69
17.29
22.22
26.33
29.50
33.33
START
1 ug/ml Aroclor 1260

DATA SUMMARY

Table I.1

SAMPLE NUMBER	MATRIX	WEIGHT	SURFACE AREA
63091	plant	4.11 g	
63091	surface		100 sq. in.
63092	plant	4.50 g	
63092	surface		100 sq. in.

DATA SUMMARY

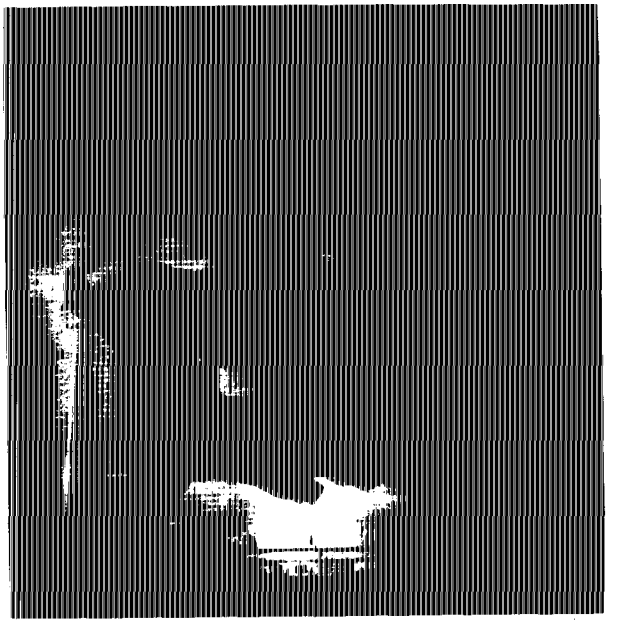
Table II.1

SAMPLE NUMBER	ug/g PCB	ug/sq. in. PCB
63091	5.0	0.04
63092	2.0	0.01

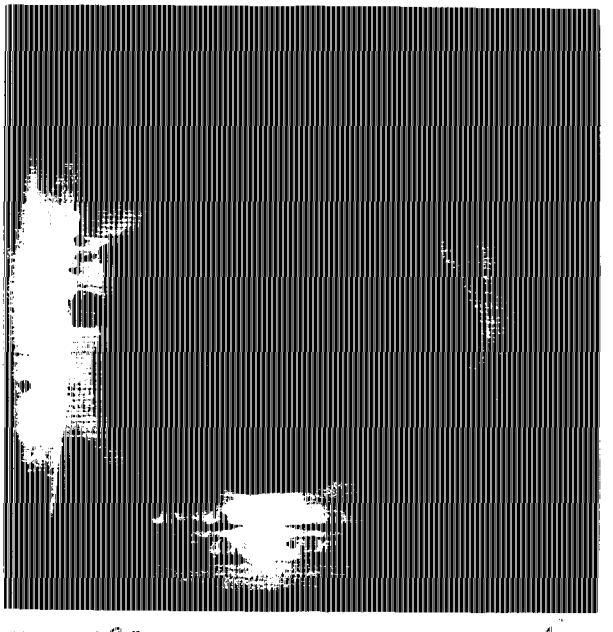
50.00 STOP
START
0.96
1.75
2.17
2.70
3.31
4.76
5.43
6.39
7.11
8.04
8.96
10.10
11.32
14.56
15.95
17.26
19.01
22.24
25.79
28.12
28.97
sample # 62091 SGB lettuce extract

START
0.56
0.84
1.20
1.41
1.86
3.64
4.76
5.46
6.39
7.14
8.09
8.99
10.09
11.34
13.12
14.50
15.91
17.31
19.86
22.22
28.13
33.02
Sample # 6X091 SBB latitude surface extract
Surface 2

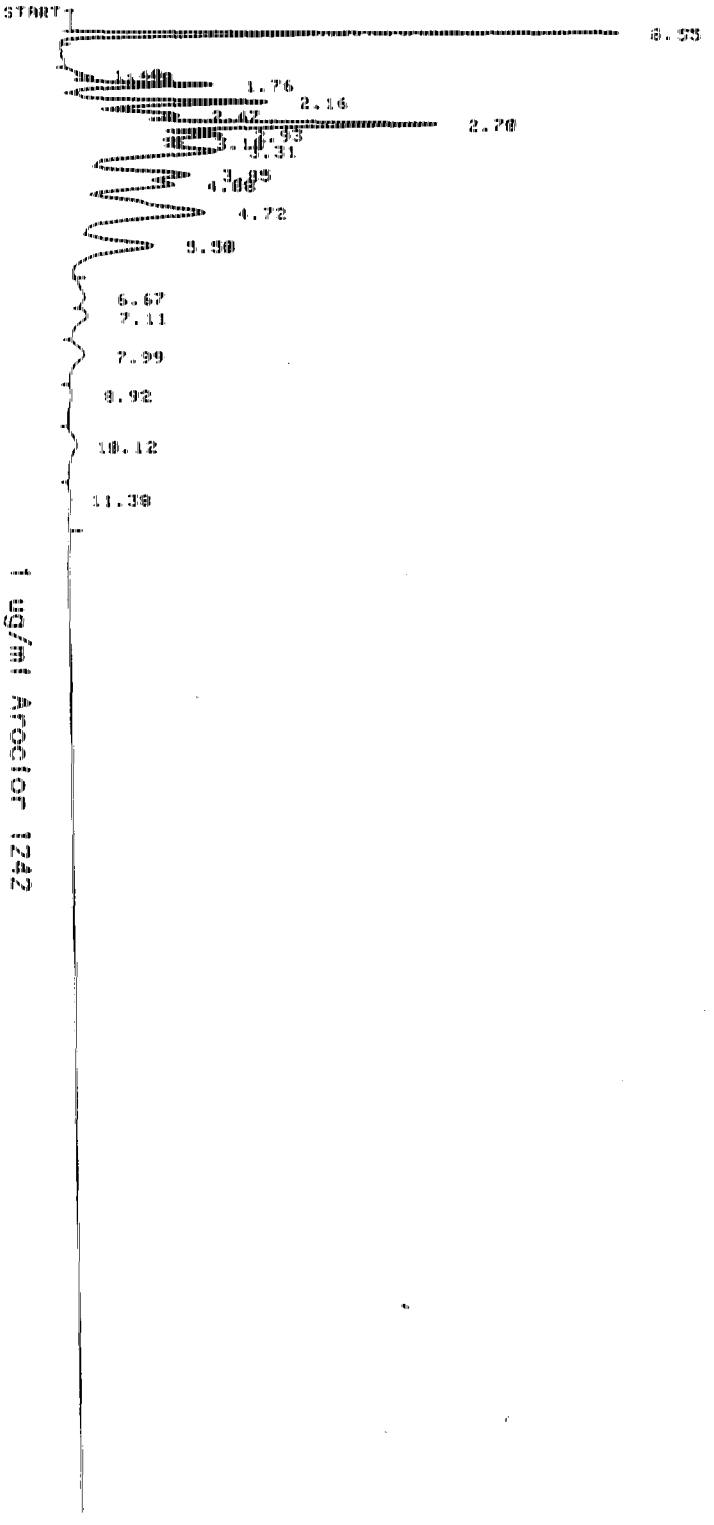
START
0.56
0.00
1.42
1.20
1.85
2.20
3.19
3.62
5.65
7.09
8.04
8.97
10.02
11.31
12.45
15.70
17.23
19.79
22.15
23.54
28.18
sample # 03002 sea lettuce surface extract
WAFFAC



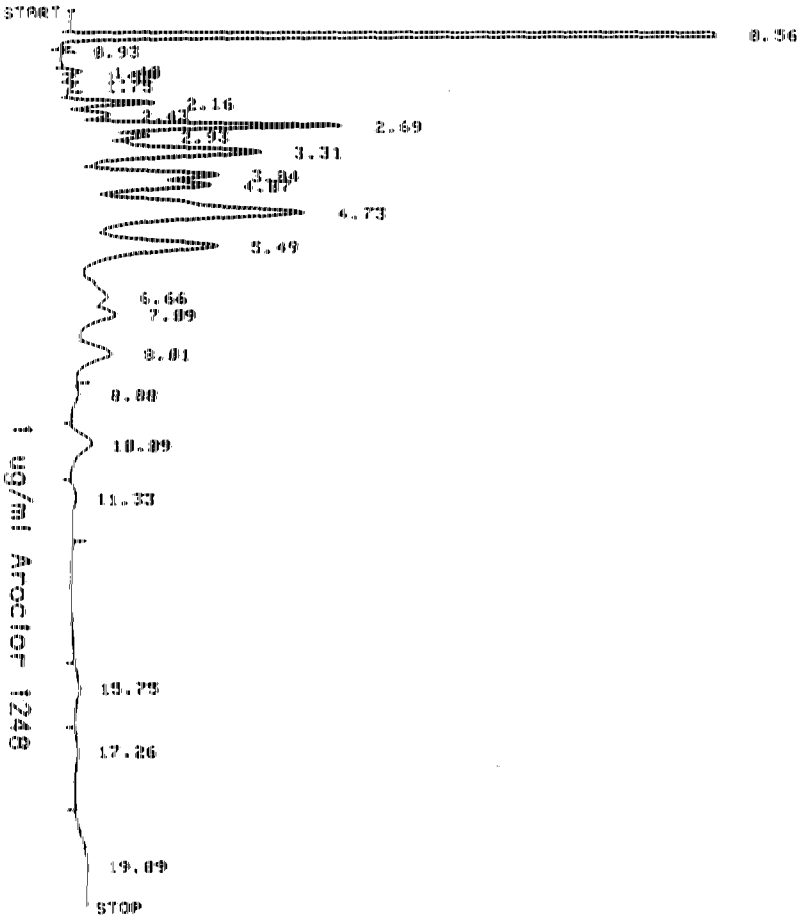
63091 representative portion



63092 representative portion



1 ug/ml Aroclor 1242



HP RUN # 112 SEP/30/82 TIME 03:42:42
ID:63091.2 BOTTLE 10
EST3

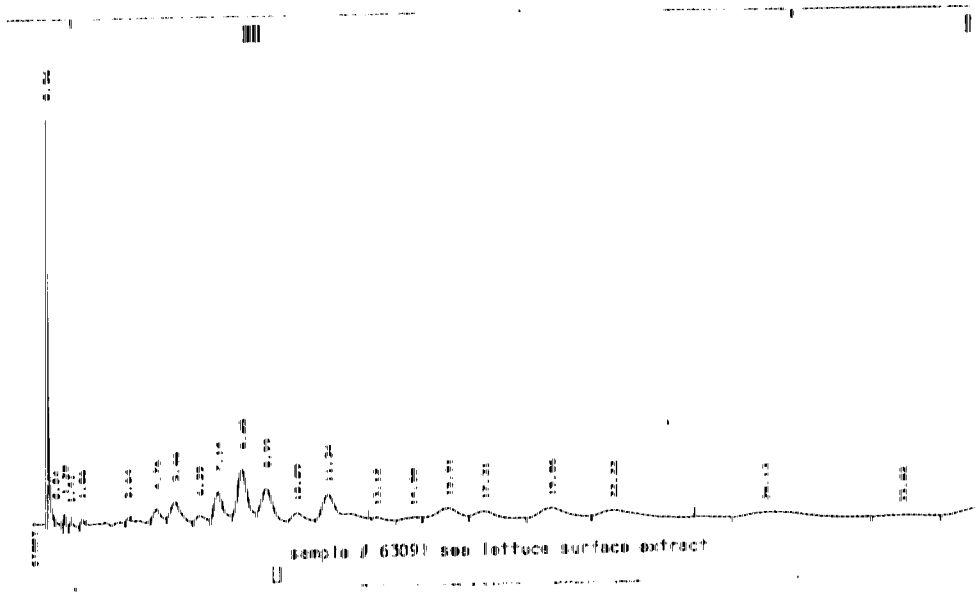
RT	EXP RT	AREA	CAL #	AMT
0.56	0.54	820800	(R) 1	664.077

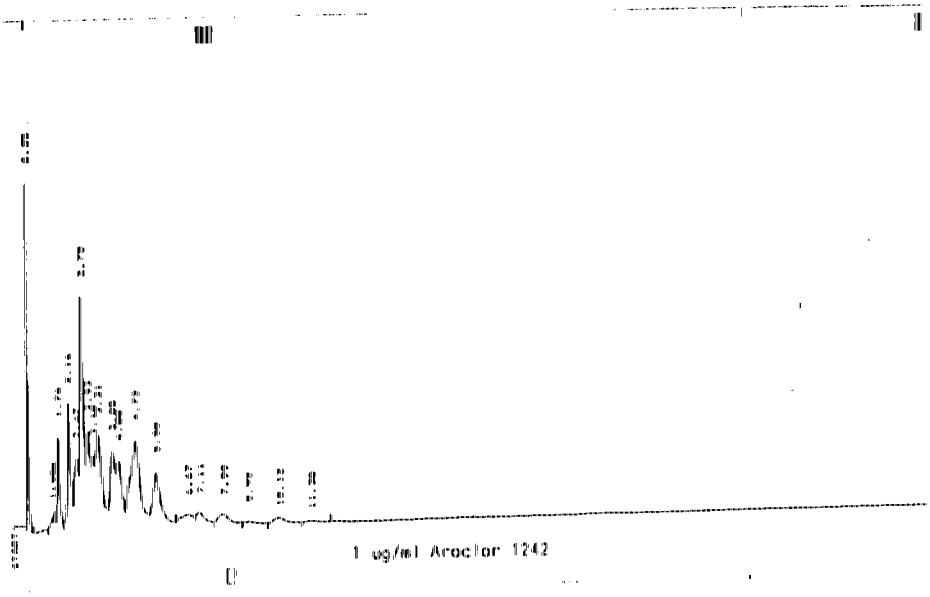
DIL FACTOR: 1.0000 E+ 0

START
0.55
1.46
1.48
1.55
2.17
2.70
3.32
4.00
5.77
5.44
6.41
7.12
8.04
8.97
10.10
11.32
12.09
13.19
14.70
16.03
17.25
22.17
29.51
33.49
sample #50092 sea lettuce extract

Sample # 6309 992 lettuce extract

sample # 63091 see lactuca extract


sample # 63091 sea lettuce surface extract


1 ug/ml Aroclor 1242

1 ug/ml Aroclor 1248
STOP

sample 10 JR3092 300 low-dose extract
2.17
2.79
2.55
7.10
10.19
11.55
13.19
14.79
16.85
17.55
22.17
23.31
23.49