2/5/99
/in the margin/
Erlen 250 mL
with glass cap
for 2 samples
and for blank

Iodine
- Fraction 1 with pre-heated L-cysteine: 0.2030 g
15 mL cyclohexane acetic acid 1:1 vol/vol poured into three 5 mL glass tubes to put a first one into oil, pipette this solution in erlen, then pour a second tube of cyclo-acetic acid into small glass tube which contained oil measured on a new scale, and pipette in erlen, and finally pour the small tube which contained the oil with 3rd tube of cyclo-acetic acid, and pipette in erlen. Then oil is accurately weighed to minimize losses.
The blank contains cyclo-acetic acid divided into 3 tubes, also to reproduce the loss of cyclo-acetic acid generated by these 3 tubes.

7.1 mL $Na_2S_2O_3$

- Fraction 1 without pre-heated L-cystein: 0.2056 g (seems less dry)

13.9 mL – 7.1 mL = 6.8 mL
- Potassium dichromate: 0.1704 g
/in the margin: to test $Na_2S_2O_3$/
Gives very very dark upon addition of KI 10% and no clear yellow color = light green
went too far?
After starch addition and titration, this gave light green and not colorless or yellow...
23 mL $Na_2S_2O_3$
N = 20.394 x 0.1704 g/23 mL = 0.15 N

- The blank gives very dark upon addition of KI, and there was a fuschia layer at the surface 41.8 mL – 15.0 mL = $Na_2S_2O_3$ --> 26.8 mL

Iodine Fraction 1 with L-cystein

$$\frac{(B-S) \times N \times 12.69}{\text{mass (g)}} = \frac{(26.8\ mL - 7.1\ mL) \times 0.15 \times 12.69}{0.2030\ g}$$

$= 184.72$

2/9/9

Iodine Fraction 1 without L-cystein:

$$\frac{(26.8\ mL - 6.8\ mL) \times 0.15 \times 12.69}{0.2056\ g} = 185.16$$

2/8/99

Extraction Krill	E. pacifica
with L-cystein	250 g fresh K.
	1,500 mL acetone
	0.1 g L-cystein
	20 min.
	Filtration
	Rince 500 ml acetone
	Filtration
	500 mL + butanol

Determination of density

Oil fraction 1 heated without L-cystein :
1 empty cup 0.9989 g
cup + oil (1 mL) 2.0554 g

2 empty cup 0.9913 g
cup + oil tip rinsing 1.0126 g

3 oil fraction heated + L-cystein:
empty cup 0.9938 g
cup + oil (1 mL) 2.0387 g

4 empty cup 0.9966 g
cup + tip rinsing 1.0410 g

Note: Do not pipette twice.
Pipette only once the oil in the same tip. Do it again!

69
2/9/99
Extraction of E-pacifica krill without L-cystein

Batch evaporation on Friday 2/5/99

t-butanol heating bath does not heat at all (not at 100% in any case, the light does not go on)
Then rely on vacuum only
not lab

Volatiles + humidity
Fraction 1 not heated without L-cysteine
Note: with significantly the same starting weight, the weight goes down less rapidly than for oil with L-cysteine.
Hypothesis: L-cysteine reduces, thus prevents oxidation, and oxidation produces volatiles (smell, L-cysteine seems to prevent the production of volatiles, thus the weight seems to go down more rapidly given the lack of these volatiles?

/in the left margin:
Can cysteine produce its ½ action (reduce) and then disappear from the medium and this action lasts?

L-cysteine seems to remain at the bottom of the oil container fraction 1 (with L-cysteine). Could this be that cysteine goes into the filtrate by gravity and does not remain in the residue or in the t-butanol fraction?

Try t-butanol and L-cysteine

2/9/99
empty cup 6.5278 g
oil only 5.0017 g

cup + oil 11.5299 g	Time 0
1.817 < 11.3213 g	Time 30 minutes
0.677 < 11.2456 g	" 1 hour
0.447 < 11.1958 g	" 1.5 hours
0.367 < 11.1552 g	" 2 hours
0.327 < 11.1192 g	" 2.5 hours
0.237 < 11.0940 g	" 3 hours

Determination of density starting with oil from the volatiles + humidity test (fraction 1) without L-cysteine (2.8 /illegible
not heated before the test)

1 empty cup: 0.9969 g
cup + oil (1 mL): 1.8125 g 2/10/99 1.8147g

2 empty cup: 0.9971 g
cup+ oil (tip rinsing): 1.0803 g 2/10/99 1.8000 g
/illegible/ it nonetheless

1 1.8125 g – 0.9969 g = 0.8156 g
2 1.0803 g – 0.9971 g = 0.0832 g

0.8156 g + 0.0832 g = 0.8988 g

thus density of fraction 1: 0.8988 g/mL

2/10/99 Extraction of krill <u>E. pacifica</u>
without L-cysteine in acetone
with 0.1 g L-cysteine in t-butanol
Evaporation of t-butanol with cysteine

2/11/99 Extraction of krill <u>E. pacifica</u>
without L-cysteine in acetone
without L-cysteine in t-butanol

2/11/99 Saponification
1x fraction 1 with cysteine (one layer at the surface like softening butter)
2x fraction 2 with cysteine (seems to have a melting point < 80ºC)
1x blank
Heated for 15 minutes at 125ºC for them to dry
in desiccator to bring back at room temperature
fraction 1 with cysteine 5.0041 g
fraction 2 with cysteine 5.0212 g
fraction 2 with cysteine 2.8323 g

fraction 1 with cysteine 17.5 mL H Cl 1N
fraction 2 with cysteine 42.0 mL – 17.5 mL = 24.5 mL HCl1N
(2.8323 g) fraction 2 with cysteine See below
10.7 + 20.4
blank (23.1 mL – 12.4 mL) + (49.3 mL – 28.9 mL) = 31.1 mL HCl */illegible/*
Note: small pieces which seem overly cooked adhering to the erlen bottom
fraction 2 5.0212 g
After heating, the blank has the same color and appearance as KOH + E7OH left for a long time on the window sill
(washing of burettes) = yellow and slightly turbid ... However, the solution was prepared on 2/3/99. Shall we prepare a fresh one each time?

--> Fraction 2 (2.8323 g)
28.4 mL pH 6

+ 1 mL KOH	then	pH 6.4
+ 1 mL KOH	"	" 6.8
+ 1 mL KOH	"	" 7.6
+ 1 drop HCl		7.2
+ 1 drop HCl		7.2
+ 1 drop HCl		7.2
+ 2 drops HCl		7.2
+ 3 drops HCl		7.2
+ 4 drops HCl		7.0

28.9 mL – 28.4 mL = 0.5 mL

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