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 3<sup>rd</sup> 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.

## $7.1 \text{ mL Na}_2\text{S}_2\text{O}_3$

- 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<sub>2</sub>S<sub>2</sub>O<sub>3/</sub>

Gives very very dark upon addition of Kl 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...

 $23 \text{ mL } Na_2S_2O_3$ N = 20.394 x 0.1704 g/23 mL = 0.15 N

- The blank gives very dark upon addition of Kl, and there was a fuschia layer at the surface  $41.8 \text{ mL} - 15.0 \text{ mL} = \text{Na}_2\text{S}_2\text{O}_3 --> 26.8 \text{ mL}$ 



Iodine Fraction 1 with L-cystein

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

= 184.72

2/9/9

Iodine Fraction 1 without L-cystein:

$$(26.8 \text{ mL} - 6.8 \text{ mL}) \times 0.15 \times 12.69 = 185.16$$

0.2056 g

2/8/99

Extraction Krill with L-cystein

E. pacifica 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

14



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

<u>Note</u>: 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



```
.19
```

2/9/99

```
empty cup 6.5278 g
oil only 5.0017 g
cup + oil 11.5299 g
                                   Time 0
1.817 < 11.3213 \text{ g}
                                   Time 30 minutes
0.677 \le 11.2456 \,\mathrm{g}
                                         1 hour
0.447 \le 11.1958 \text{ g}
                                         1.5 hours
0.367 \le 11.1552 \text{ g}
                                         2 hours
                                     " 2.5 hours
0.327 < 11.1192 g
                                     " 3 hours
0.237 \le 11.0940 \text{ g}
```

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 g2 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

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## 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 < 80°C)

1x blank

Heated for 15 minutes at 125°C 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 **~** 7.6 + 1 mL KOH " + 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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