DISTILLATION
as a step in tritium analysis

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Tritium (H-3)

**ENVIRONMENTAL MONITORING**

- DATING
- hydrogeology
- oceanography
- lower limits of detection
- smaller uncertainties

**ideal tracer**
Sample preparation by electrolytical enrichment

1st distillation ➔ Electrolytical enrichment ➔ 2nd distillation
\[ A_T = \frac{N_{SA} \cdot A_{ST} \cdot D}{N_{ST} \cdot Z_I} \]

- \( N_{SA} \): net count rate of the sample (cpm)
- \( N_{ST} \): net count rate of the standard (cpm)
- \( A_{ST} \): activity concentration of the standard (Bq/kg)
- \( Z_I \): tritium enrichment factor for the given sample
- \( D \): decay correction

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<th>Step</th>
<th>Description</th>
<th>Symbol</th>
<th>Sources of uncertainty</th>
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<td>Possibility of contamination for samples with low tritium content</td>
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<td>W_{CE}, W_{CI}, W_{I}</td>
<td>Uncertainty of electronic balance, buoyancy forces, mass loss due to gas production in chemical reaction</td>
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<td>Uncertainty of amperehour-meter, current leaks</td>
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<td>D</td>
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<td>A_{T}</td>
<td>Propagation of uncertainties associated with steps 2-11</td>
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1st distillation

Electrolytical enrichment

2nd distillation

LSC spectrometry

Criteriums for pH, conductivity?
Distillation to the dry end?
Fast, slow distillation?

DIFFERENT LABORATORY PRACTICES!
1st distillation

**SAMPLE**  
500 mL of deionized water + spike

**EXPERIMENT**  
FAST distillation: the maximum power of heaters; 26 fractions  
SLOW distillation: boiling on minimum; 28 fractions
2nd distillation

SAMPLE
10 L of deionized water + spike for 20 x 500 mL subsamples

EXPERIMENT
Electrolytic run: 1024 Ah
Current: 4 A
Initial sample: 500.5 ± 0.3 mL
Final sample: 117.6 ± 0.5 mL
Distillation: 6 or 7 consecutive fractions

Average pH of fractions

"speed" 2 of distillation not under strict control!
**2nd distillation**

**SAMPLE**
10 L of deionized water + spike for 20 x 500 mL subsamples

**EXPERIMENT**
Electrolytic run: 1400 Ah (as usual routine procedure)
Current: 3.2 – 9.9 A
Initial sample: 499.1 ± 0.3 mL
Final sample: 19.9 ± 1.6 mL
Distillation: fast and slow (10 samples of each)

**U-test**
| fast - real value | 1.89 | ATTENTION! |
| slow - real value | 0.63 | OK |
| fast – slow      | 1.29 | OK |

pH measurement of 3 „cumulative“ fractions

**Graph**
- pH vs. mass % of cumulative fraction
- Data points for fast and slow distillation
- Fast distillation: 1.89 (ATTENTION!), real value: 0.63, 1.29 (OK)
- Slow distillation: 90, 85, 80, 75, 70

**Notes**
- ATTENTION!
**pH and efficiency**

**SAMPLE**
- deionized water with different pH + spike

**EXPERIMENT**
- different sample: scintillator ratio
- time-dependent measurements

Average of 7 to 9 measurements in 1 – 5 days
Counting time of each measurement: 100 min
Conclusions

BE CAREFUL WITH DISTILLATION!

„speed of distillation → pH → counting efficiency"

wrong result or at least big uncertainty!

„slow“ distillation
„calibration“ of heaters
always the same portion of residue in distillation process
control of pH
THANK YOU!