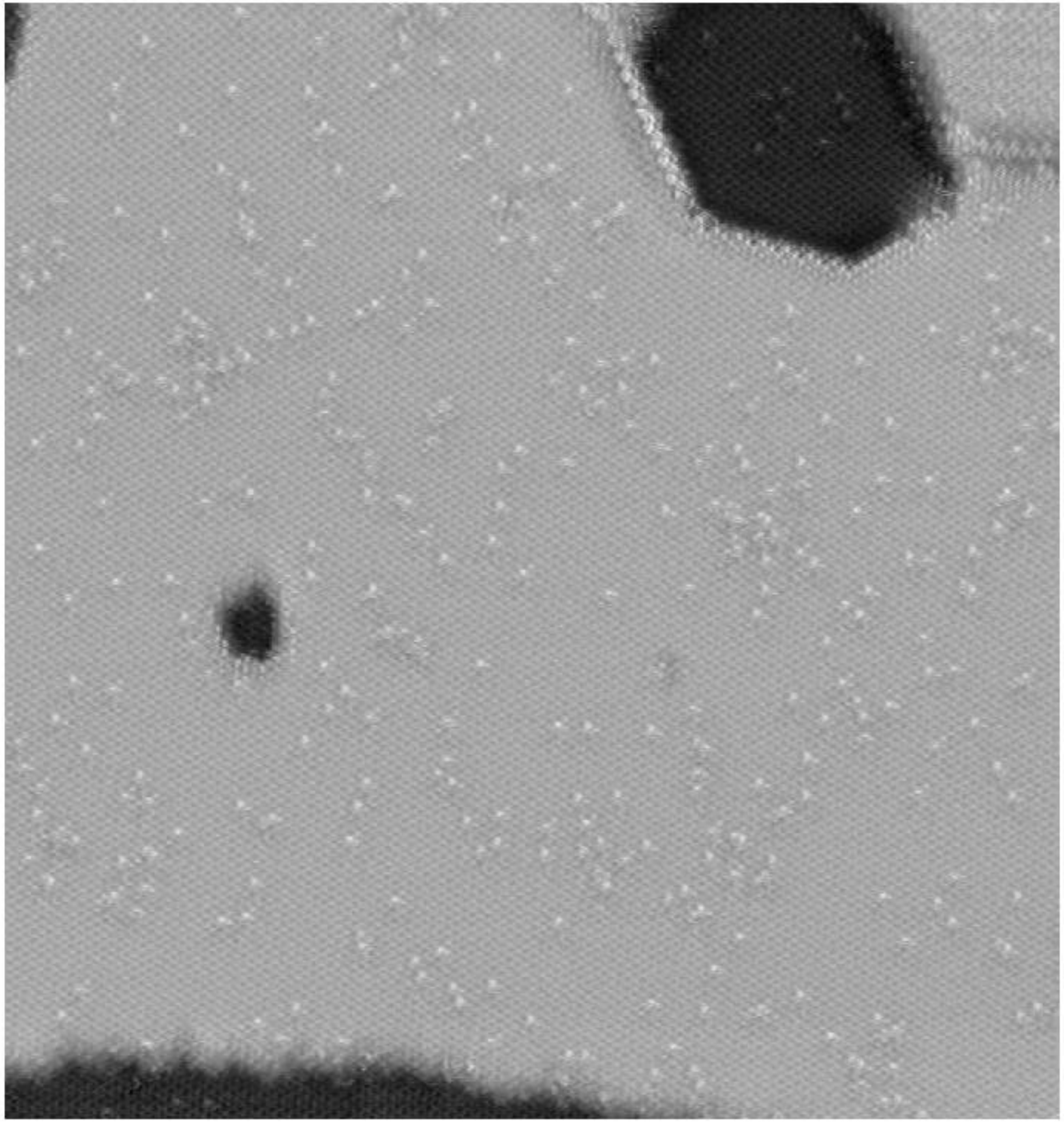
Studying SAM Growth Vial Leak

Juan Zuniga

Advisor: Lloyd Bumm

The Surface and Molecular Interface Group

- **Studies condensed phase systems**
- **STM imaging of self-assembled monolayers (SAM)**
- **Uses metal vial device to grow SAMs**



SAM Growth Vial

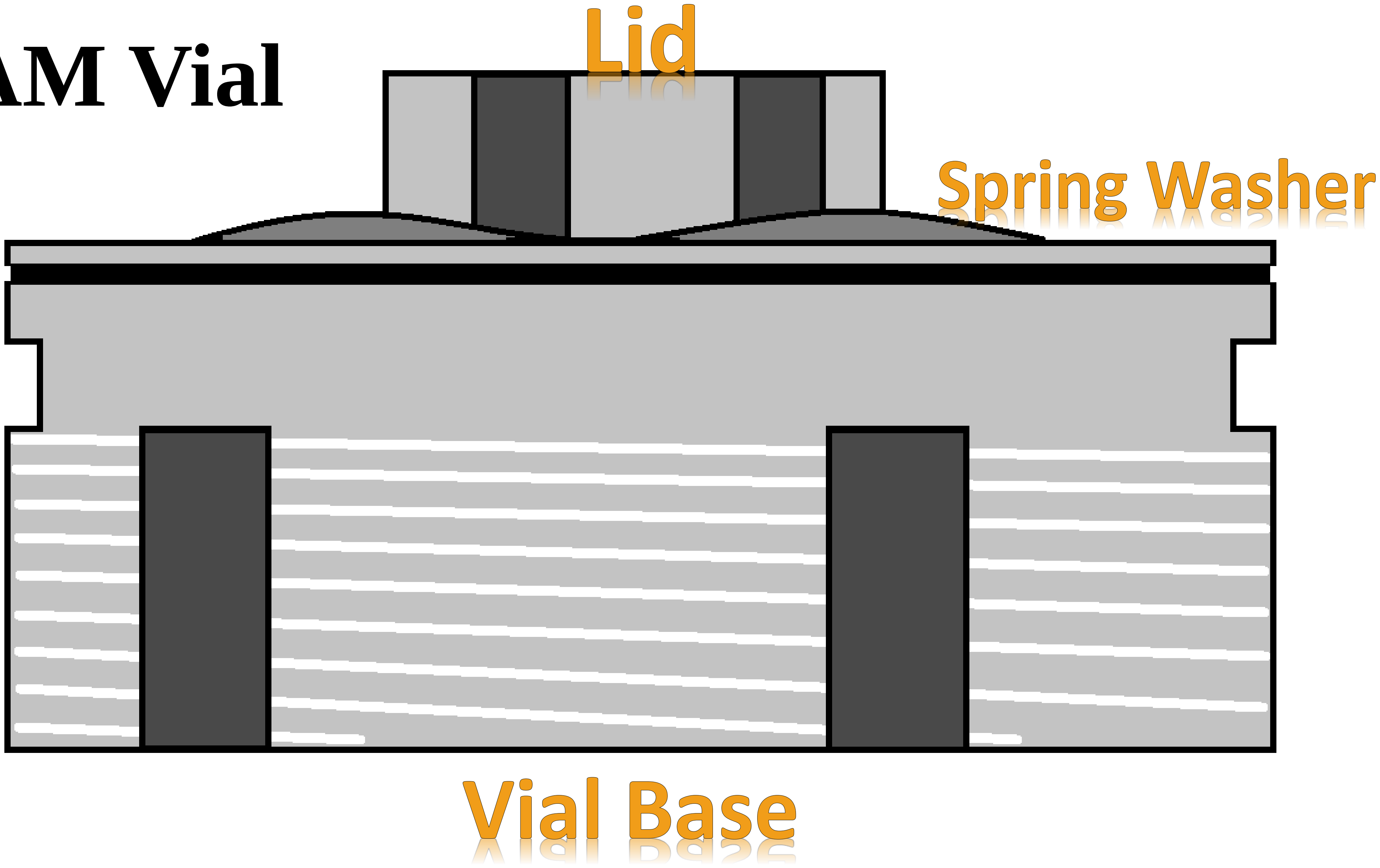
Motivation

- Vials used for creation of self-assembled monolayers.
- Vials tend to leak through o-ring seal
- Substance concentration may not remain at desired level
- SAM growth likely depends on concentration

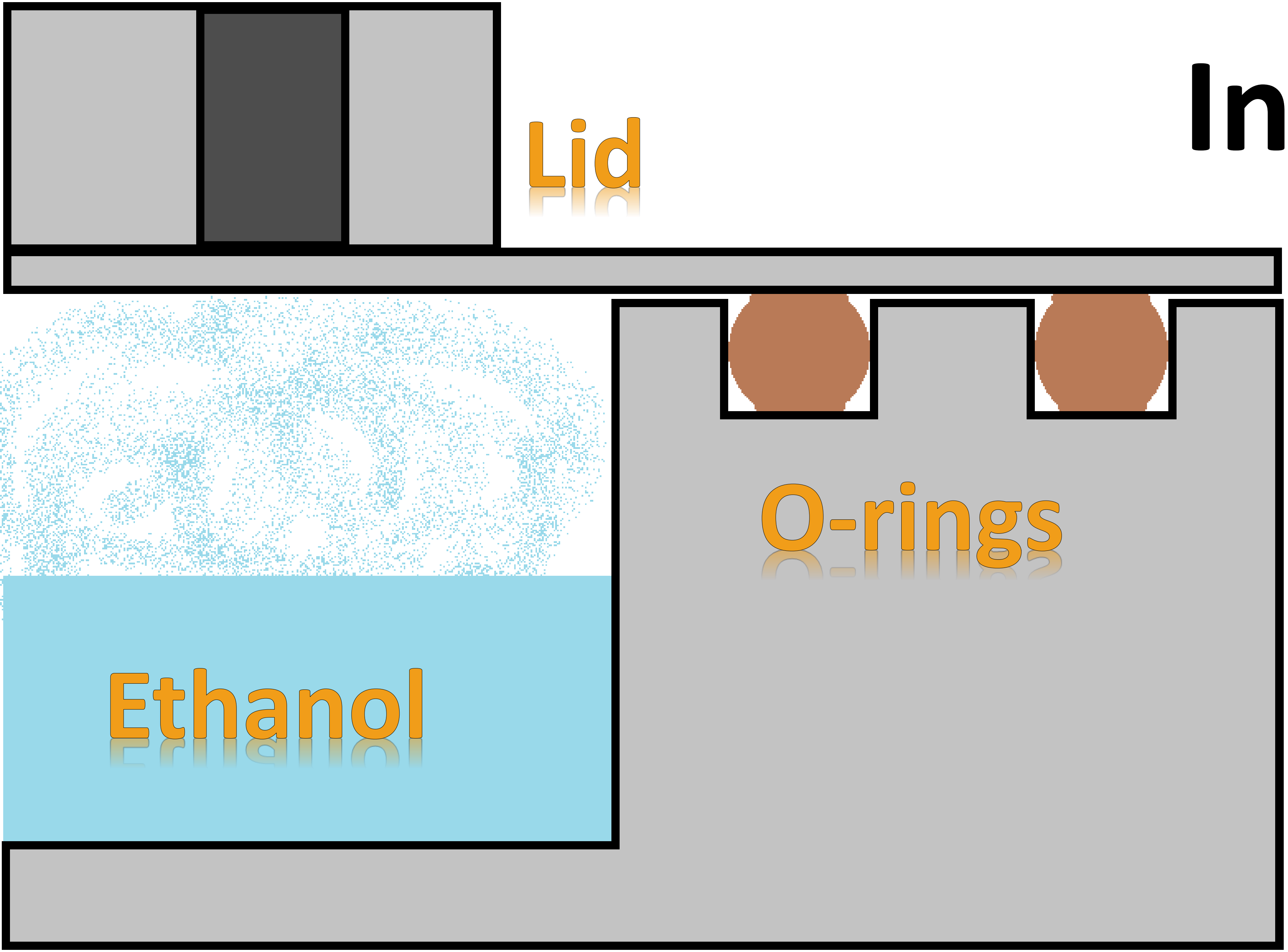
Purpose

Ascertain vial leak rates

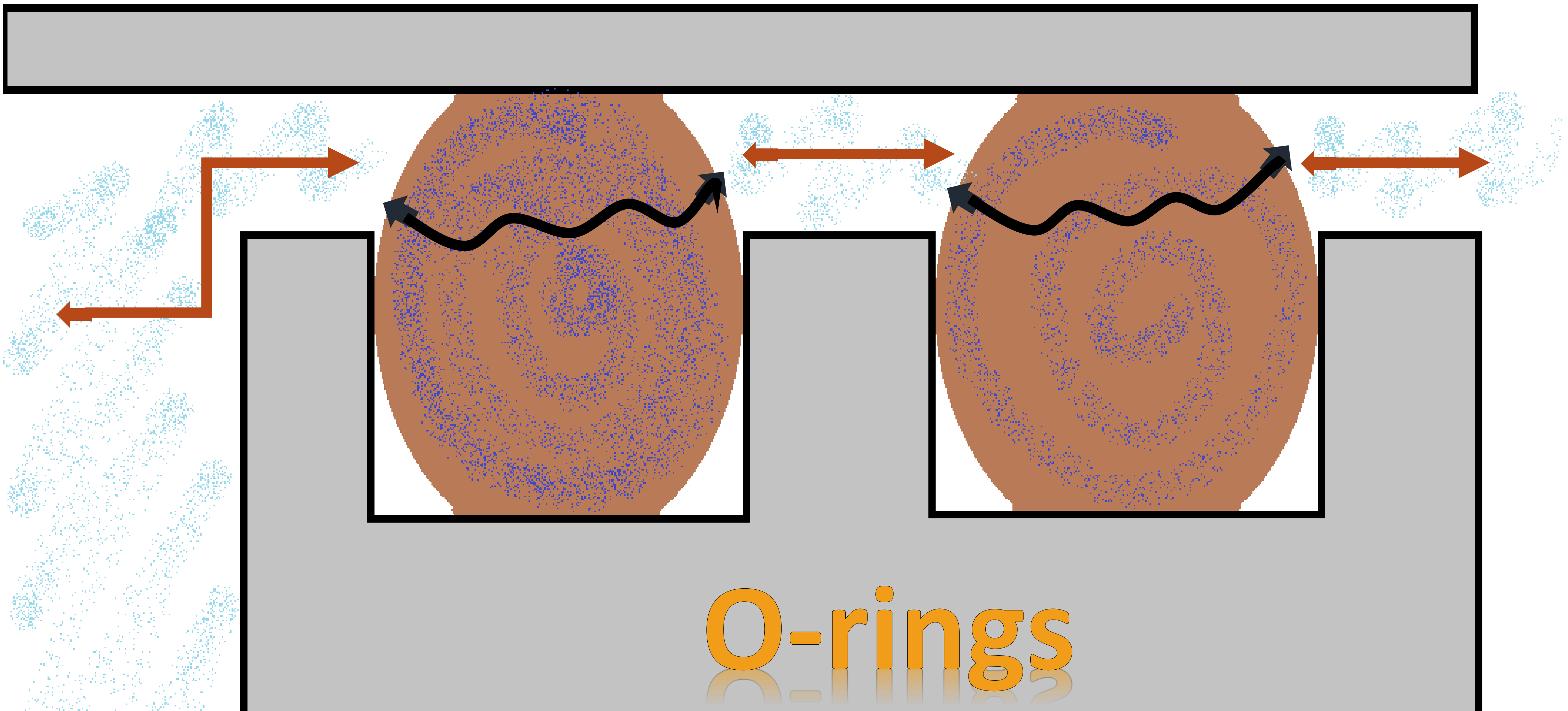
SAM Vial



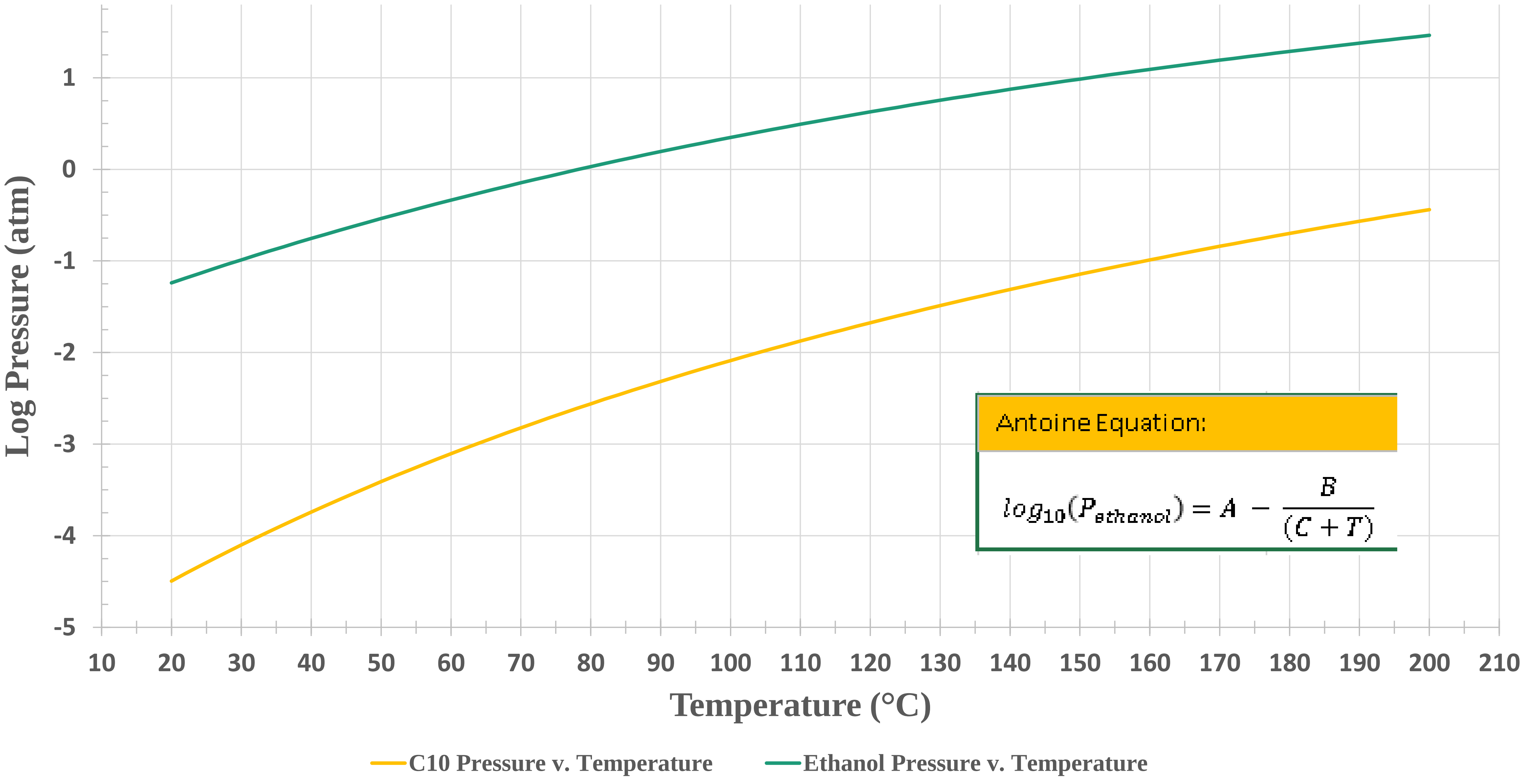
Inside Vial



Permeation Mechanism

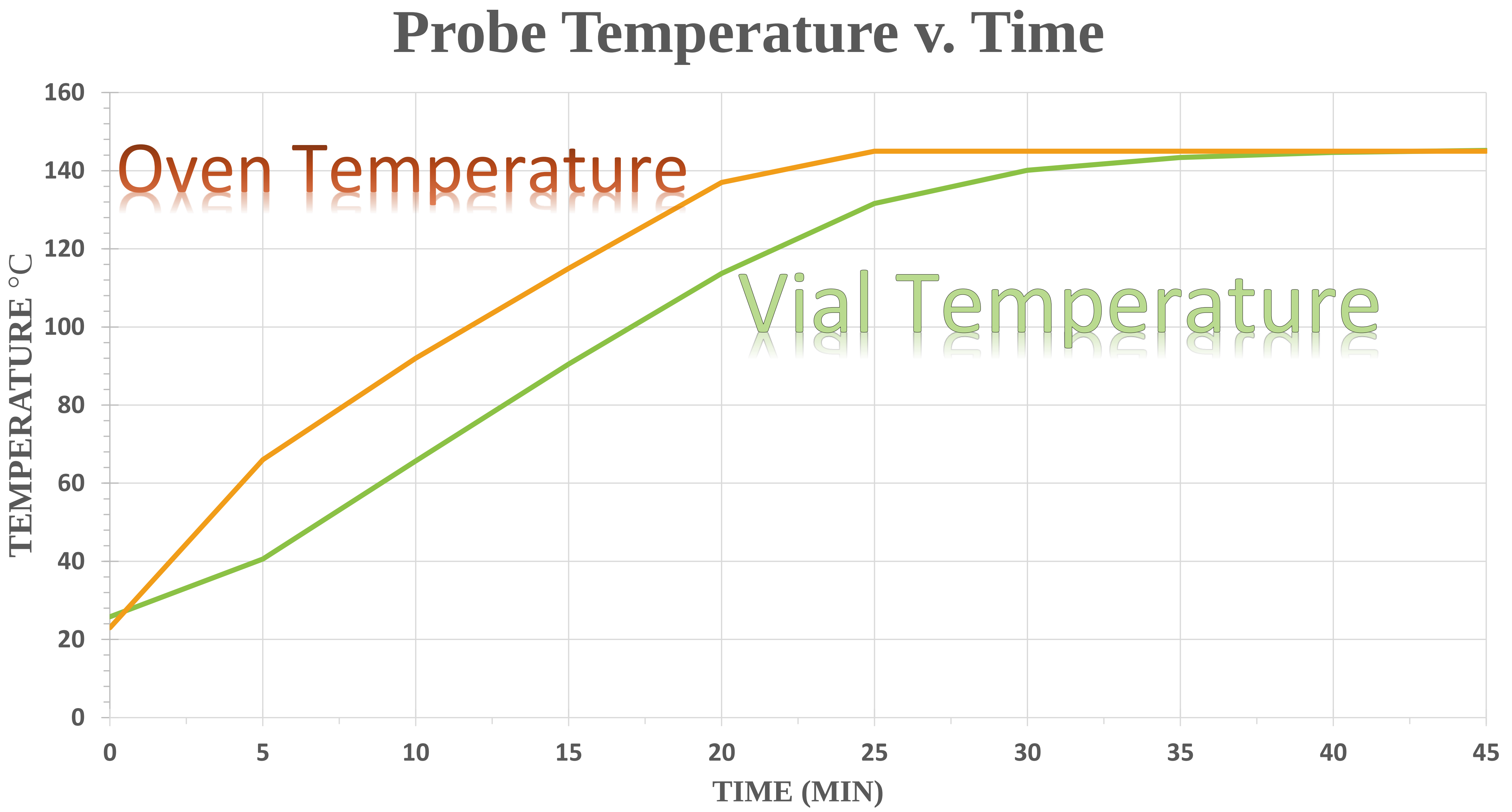


Log C10/Ethanol Vapor Pressure v. Temperature




HH309A
OMEGA
DATA LOGGER THERMOMETER
T1-T2
SETUP
THERMOCOUPLE THERMOMETER
116 C
OMEGA
ENGINEERING, INC.
MODEL 115 KC
OMEGA
OMEGA CN9000A
°C
47

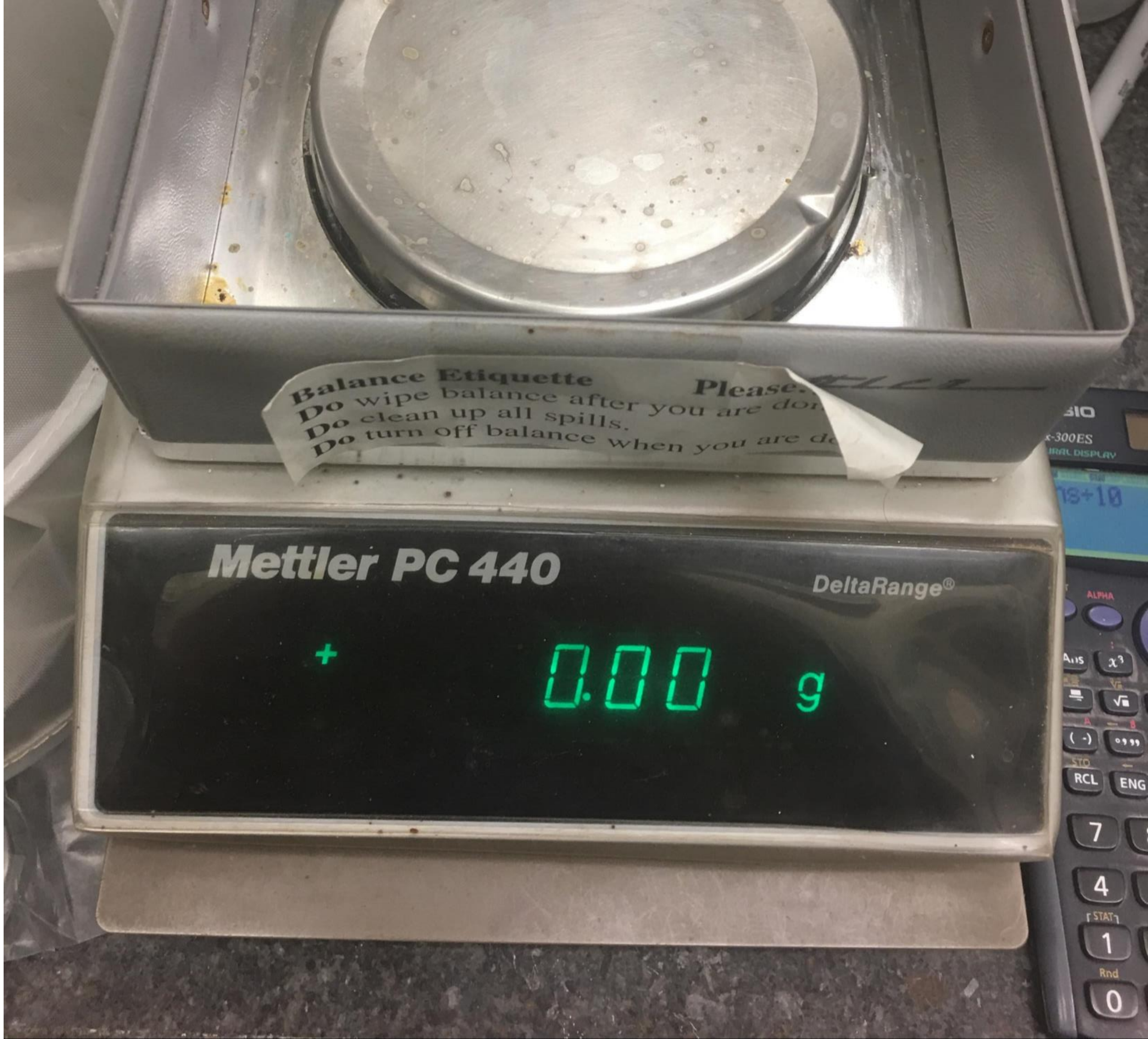

STABIL-THERM® GRAVITY OVEN
OFF
ROTATE RIGHT OR LEFT
POWER SELECTOR
RESET
OVERTEMPERATURE PROTECTION
HEATER
MAIN
MEDIUM
TEMPERATURE CONTROL
BLUE M
BLUE M Electric Company



Probe Temperature v. Time
Oven Temperature
Vial Temperature
TEMPERATURE °C
160
140
120
100
80
60
40
20
0
0
5
10
15
20
25
30
35
40
45
TIME (MIN)

Mettler PC 440 Scale

- **Maximum Weight Limit: 228 g**
- **Accuracy: 0.01 g**
- **Not so precise**
- **Standard Deviation: ~±40 mg**

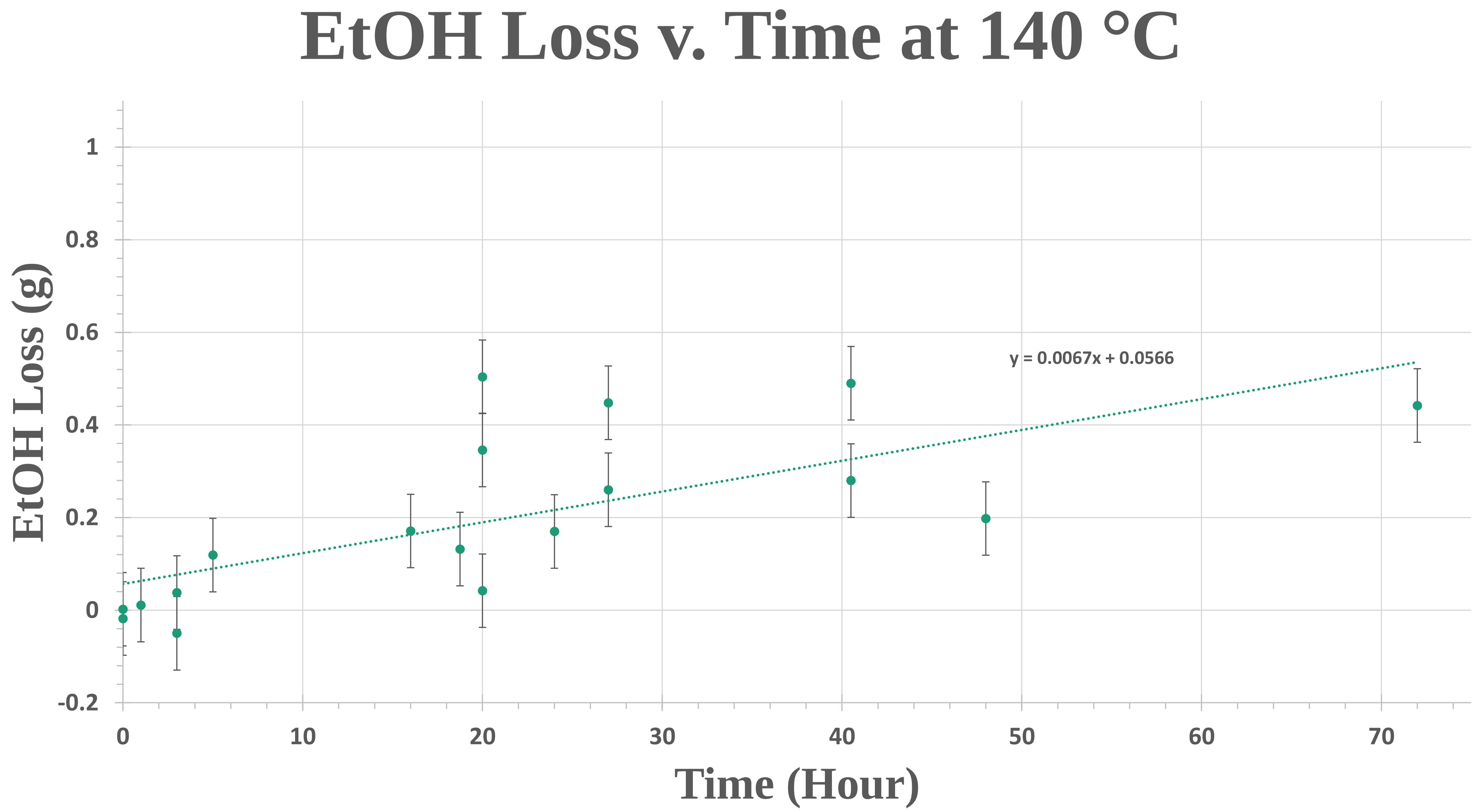


Mettler H54AR Balance

- **Maximum Weight Limit: 160 g**
- **Accuracy: 0.1 mg**
- **Precise and accurate**

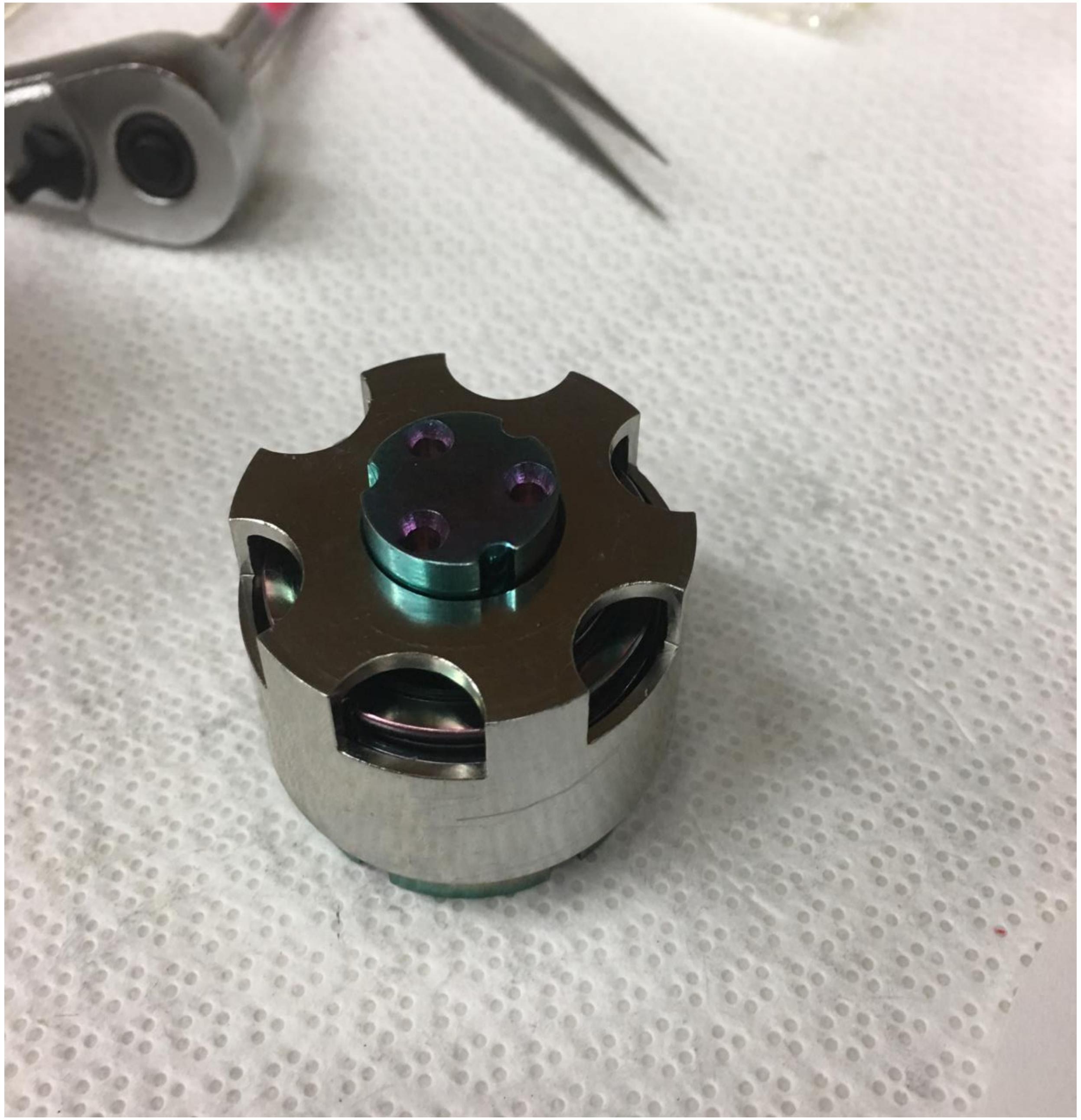
Set at 140 °C

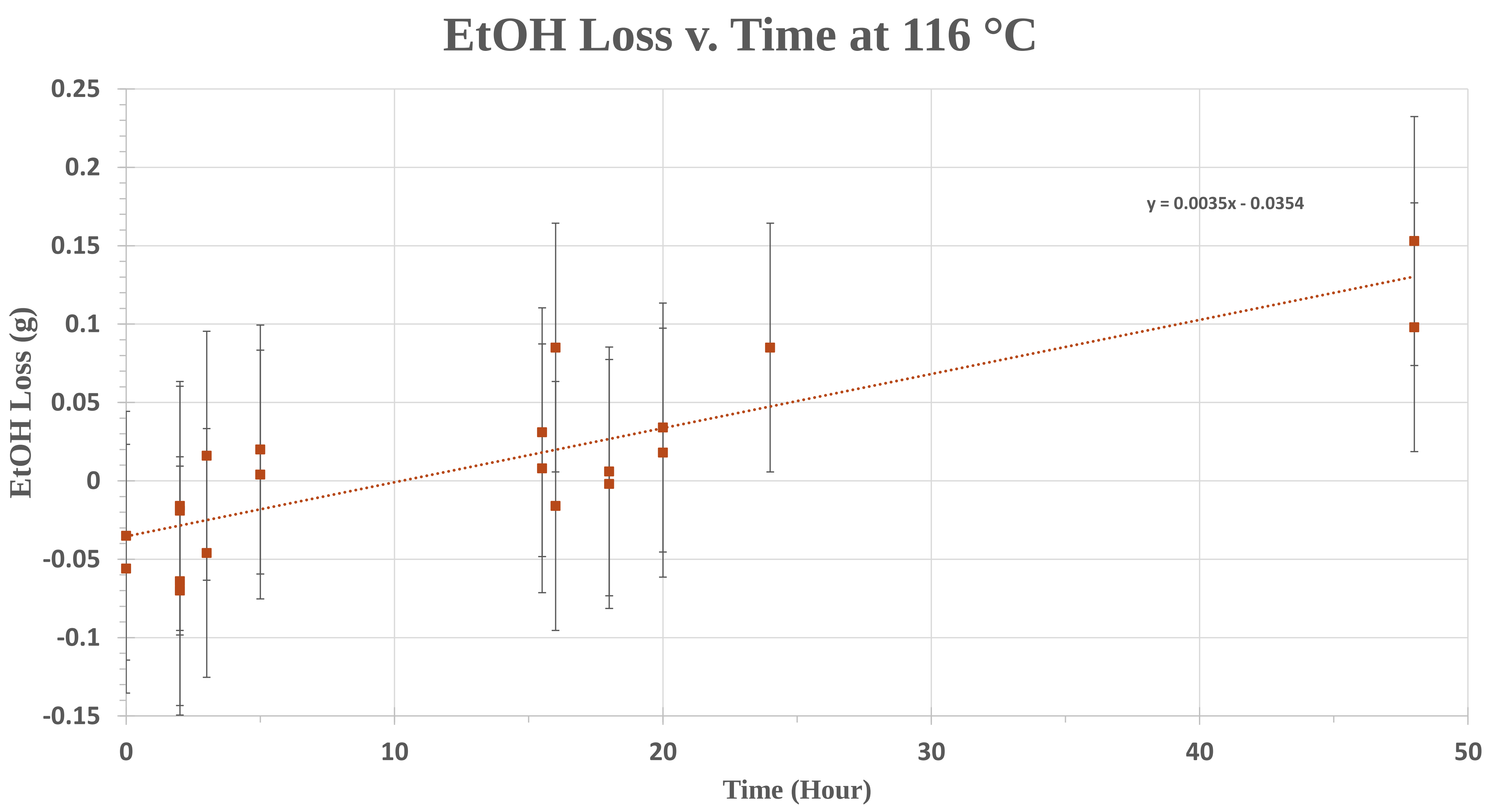
- 1.5 g EtOH
- Placed in the oven at 140 °C
- 7.5 atm of vapor pressure


EtOH Loss v. Time at 140 °C
y = 0.0067x + 0.0566
EtOH Loss (g)
Time (Hour)
1
0.8
0.6
0.4
0.2
0
-0.2
0
10
20
30
40
50
60
70

Set at 116 °C

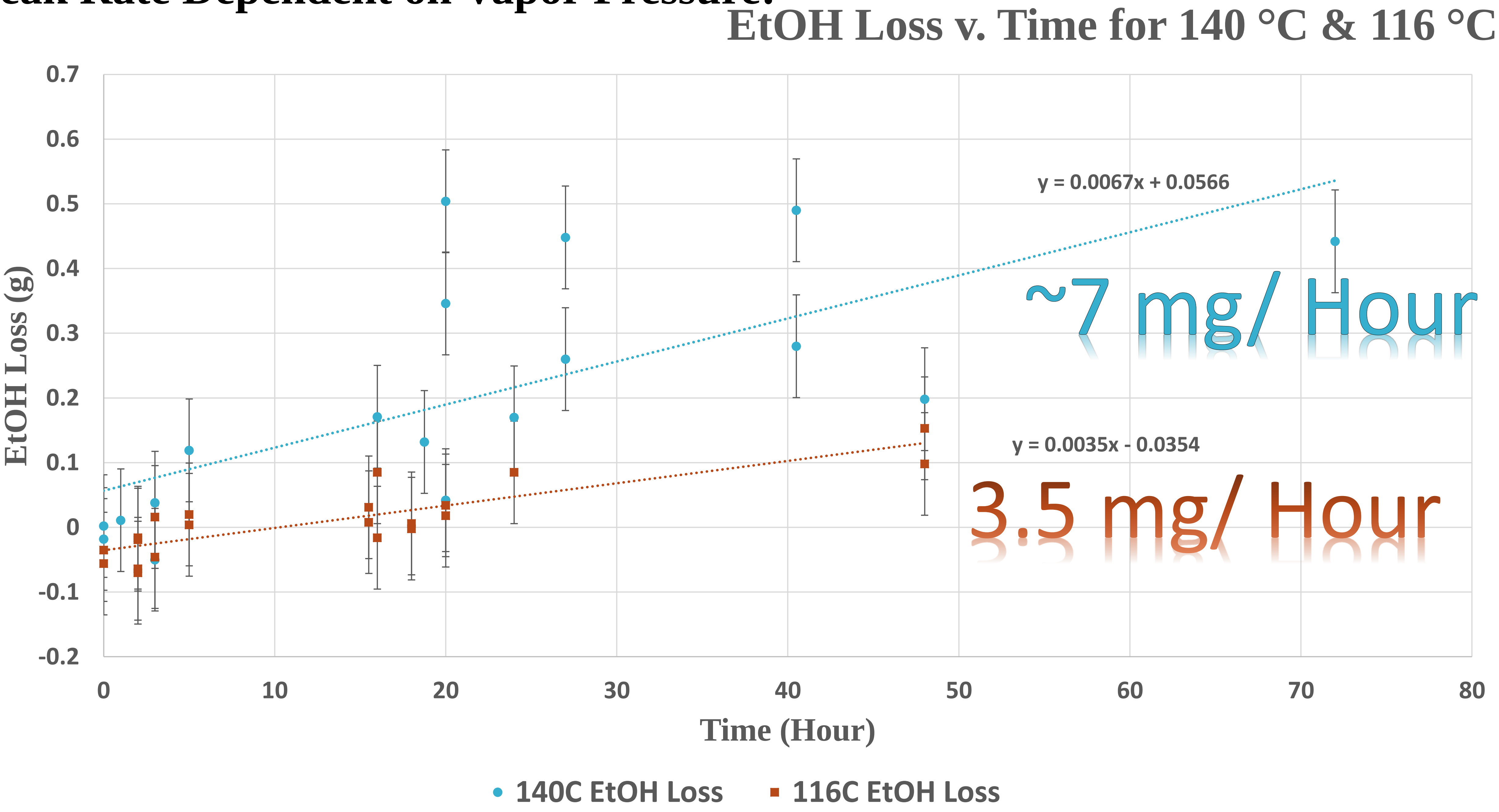
- 116 °C
- 1.5g ethanol
- 3.4 atm
- Placed in oven at variable time





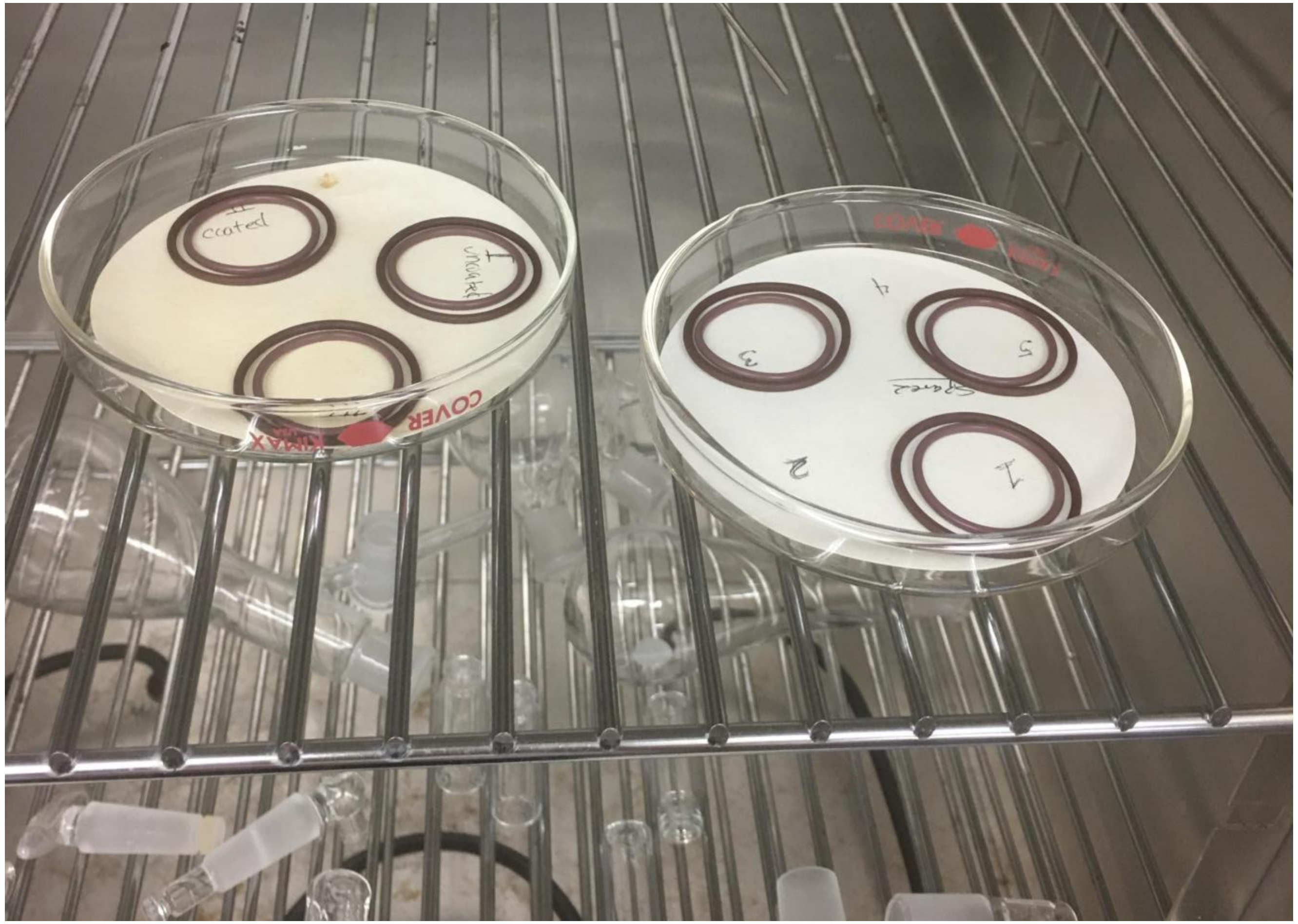
EtOH Loss v. Time at 116 °C
y = 0.0035x - 0.0354
EtOH Loss (g)
0.25
0.2
0.15
0.1
0.05
0
-0.05
-0.1
-0.15
0
10
20
30
40
50
Time (Hour)

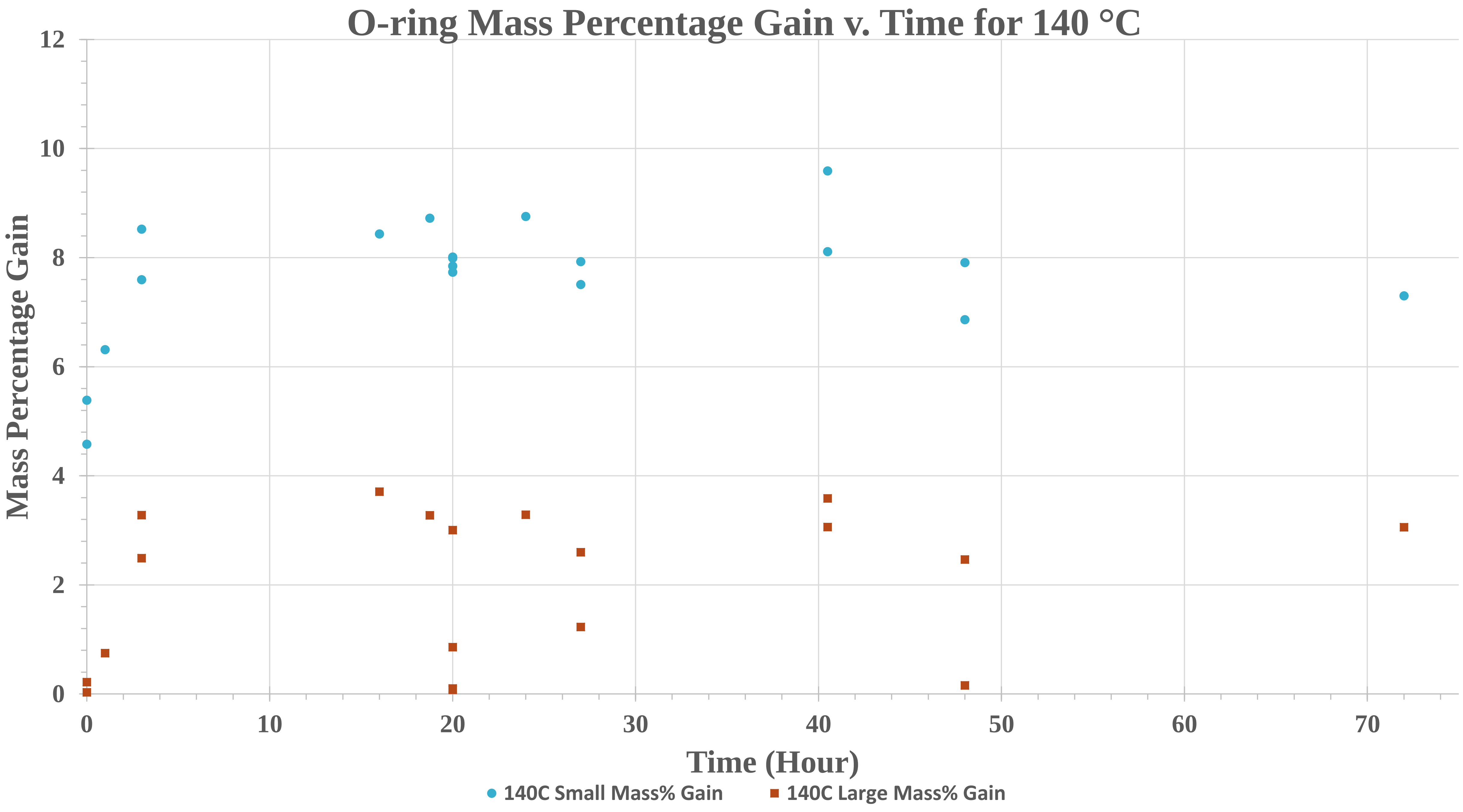
Leak Rate Dependent on Vapor Pressure!



O-ring Ethanol Absorption

- **Sets at 116 °C & 140 °C**
- **O-rings measured before and after placing in vial**
- **O-rings measured again after cooking in the oven**
  - **O-rings returned to original mass**
- **Weight difference is due to ethanol permeation**




O-ring Mass Percentage Gain v. Time for 140 °C
Mass Percentage Gain
Time (Hour)
0
2
4
6
8
10
12
0
10
20
30
40
50
60
70
140C Small Mass% Gain
140C Large Mass% Gain

Fitting Data to a Model

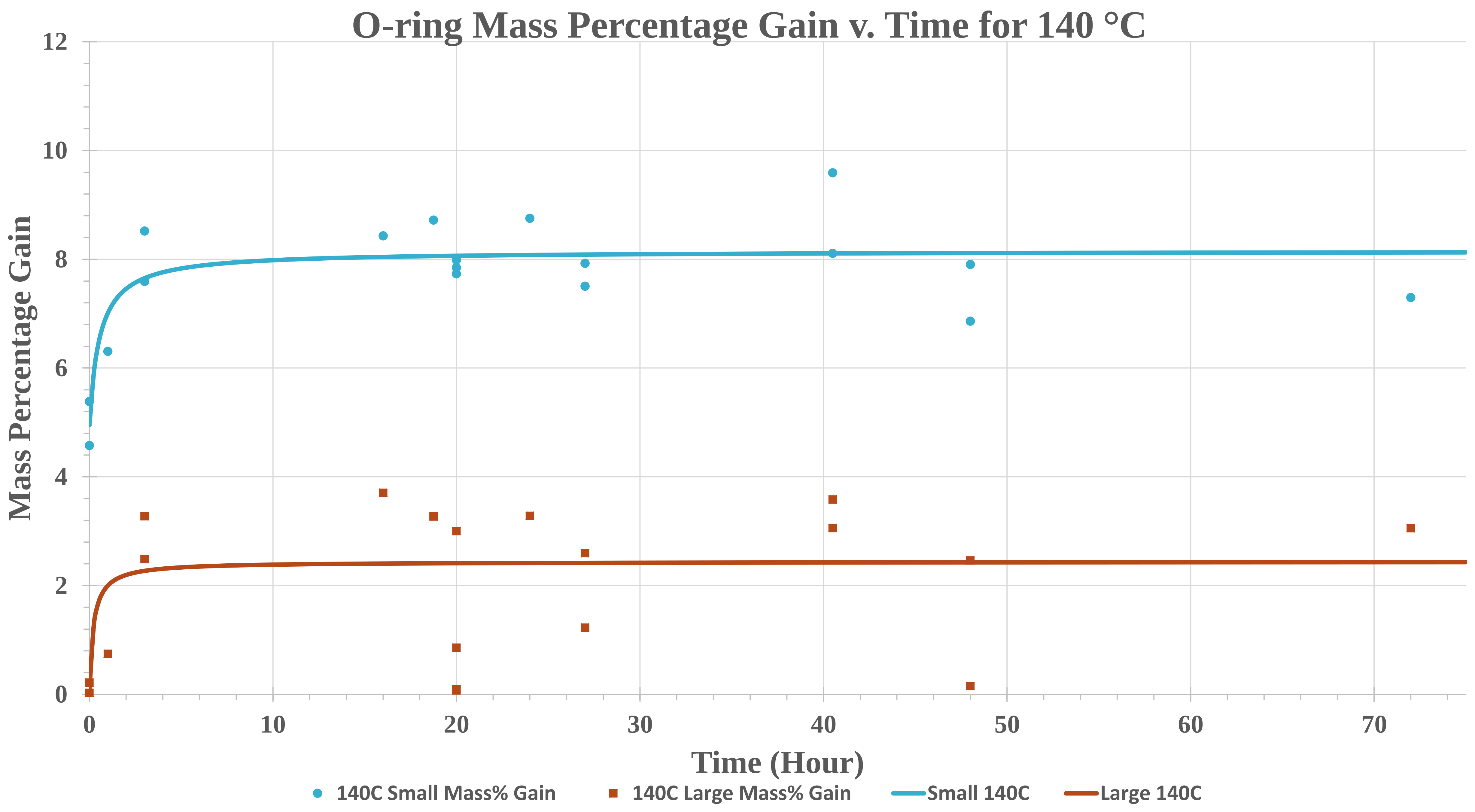
$$Abs = \frac{k_1 t}{k_2 + t} + k_3$$

At Low Times:

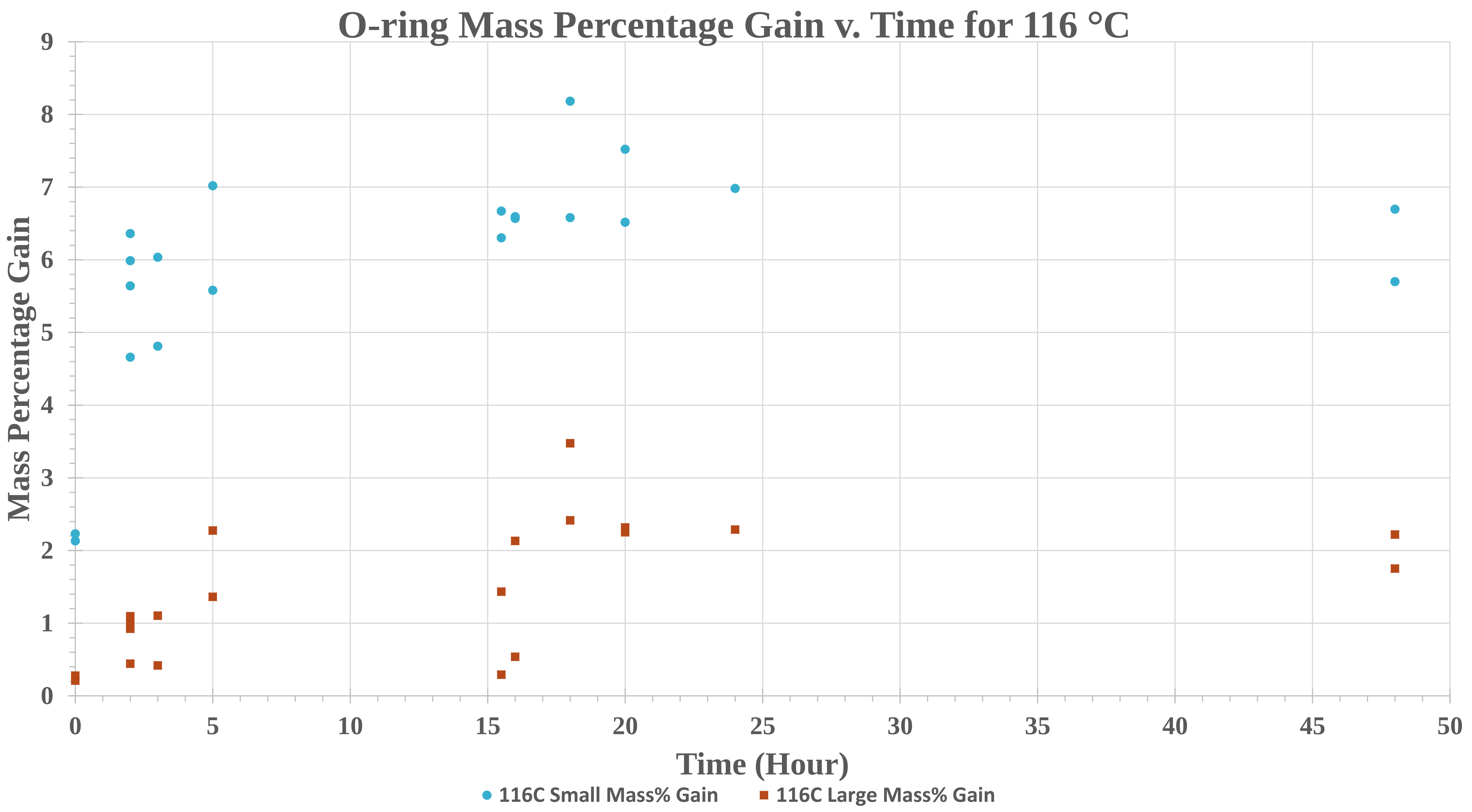
$$Abs = \frac{k_1}{k_2} t + k_3$$

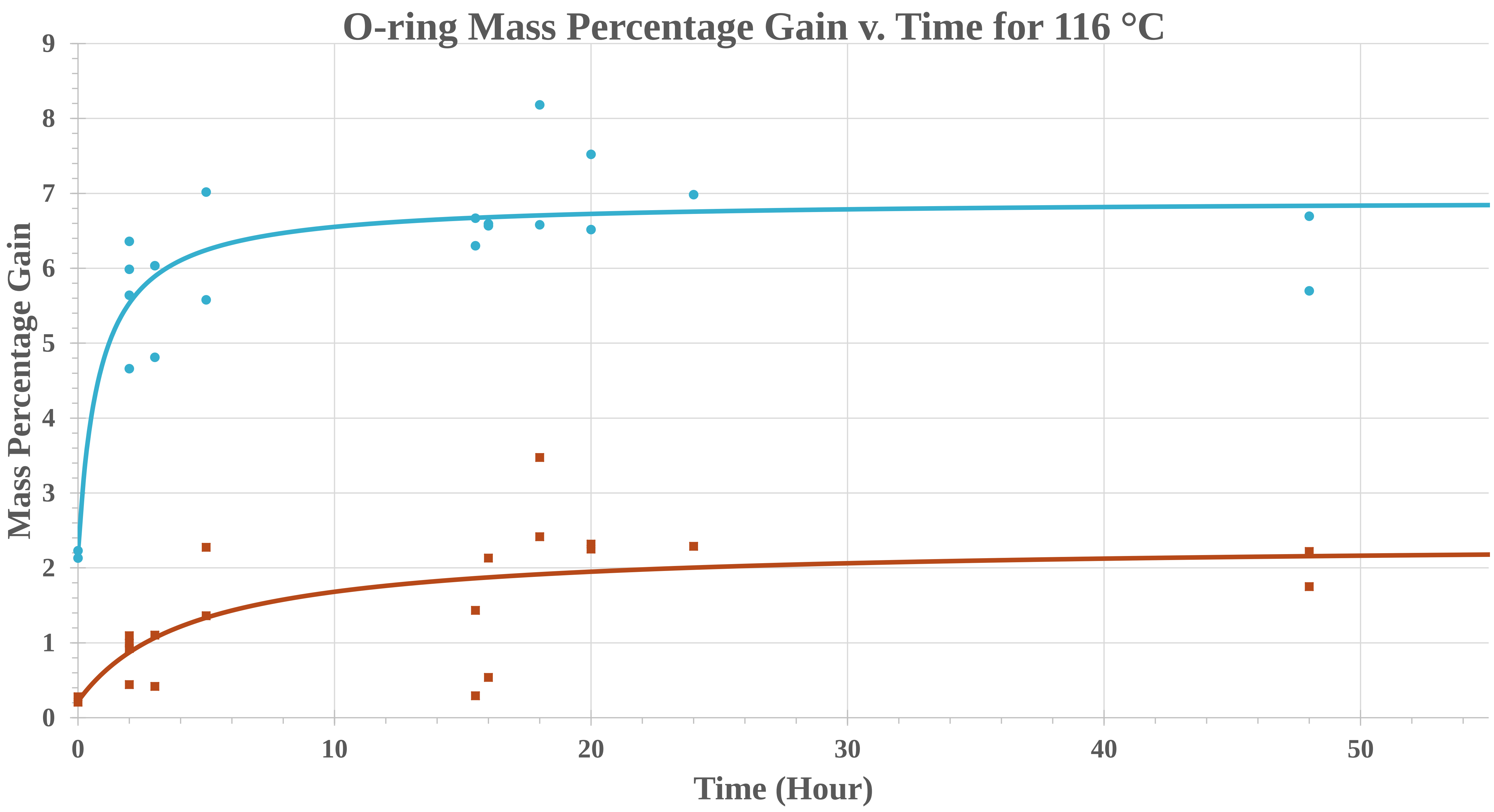
At High Times:

$$Abs = k_1 + k_3$$

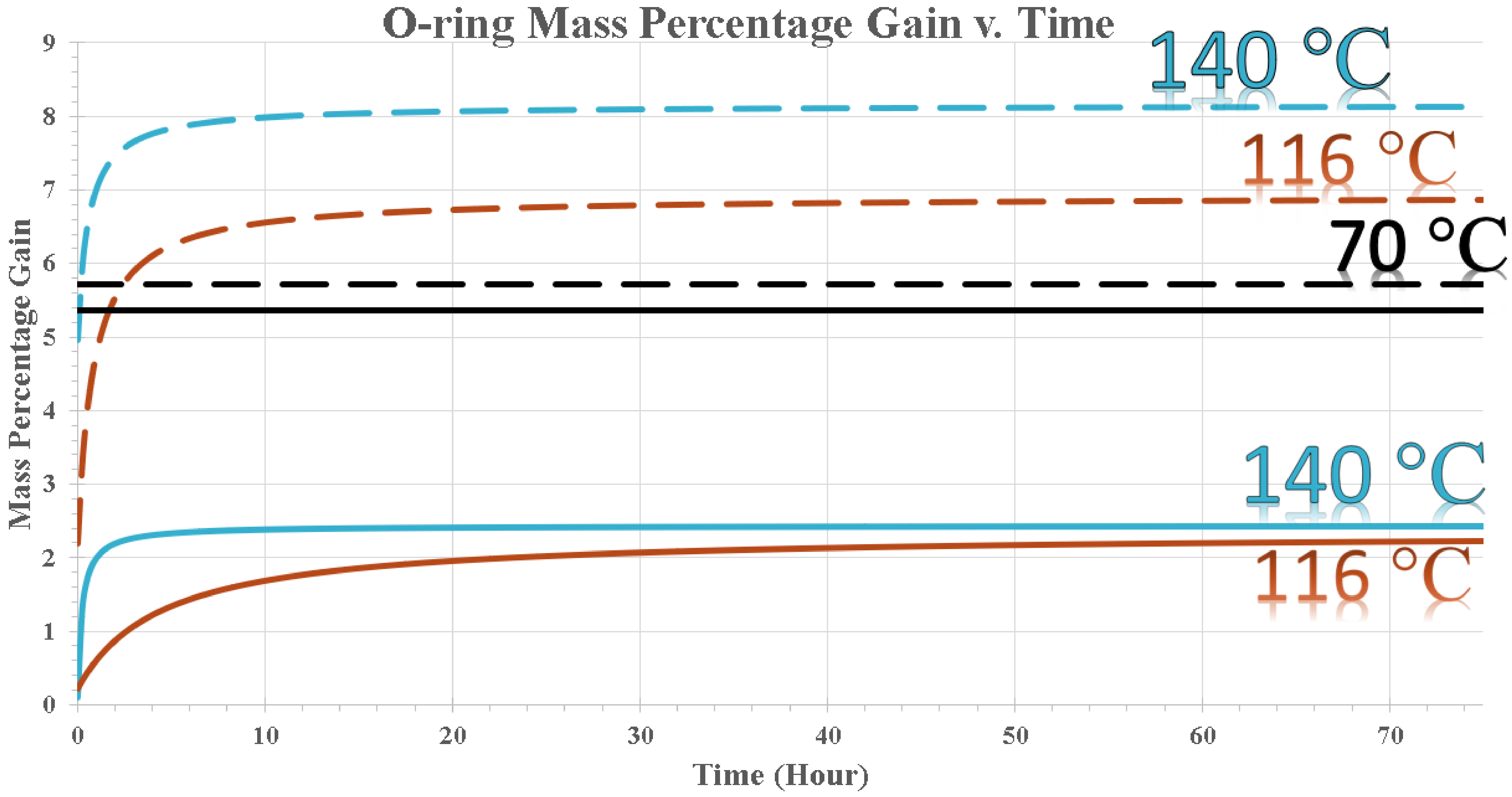


O-ring Mass Percentage Gain v. Time for 140 °C
Mass Percentage Gain
12
10
8
6
4
2
0
0
10
20
30
40
50
60
70
Time (Hour)
140C Small Mass% Gain
140C Large Mass% Gain
Small 140C
Large 140C


O-ring Mass Percentage Gain v. Time for 116 °C
Mass Percentage Gain
0
1
2
3
4
5
6
7
8
9
0
5
10
15
20
25
30
35
40
45
50
Time (Hour)
116C Small Mass% Gain
116C Large Mass% Gain


O-ring Mass Percentage Gain v. Time for 116 °C
Mass Percentage Gain
Time (Hour)
0
1
2
3
4
5
6
7
8
9
0
10
20
30
40
50

Comparing O-ring EtOH Absorptions at Different Temperatures


O-ring Mass Percentage Gain v. Time
140 °C
116 °C
70 °C
140 °C
116 °C
Mass Percentage Gain
0
1
2
3
4
5
6
7
8
9
0
10
20
30
40
50
60
70
Time (Hour)

Fick's First Law

$$J = -DS\frac{(p_2 - p_1)}{\delta}$$

$$\frac{\partial \varphi}{\partial x} = \frac{S(p_2 - p_1)}{\delta}$$

Conclusions

- Vial leak rate's likely directly proportional to vapor pressure
- O-ring ethanol absorption/saturation depends on temperature and pressure
- A less permeable material is best to prevent vial leakage

Acknowledgements

- Dr. Lloyd Bumm
- Mitchell P. Yothers
- Soumya Bhattacharya