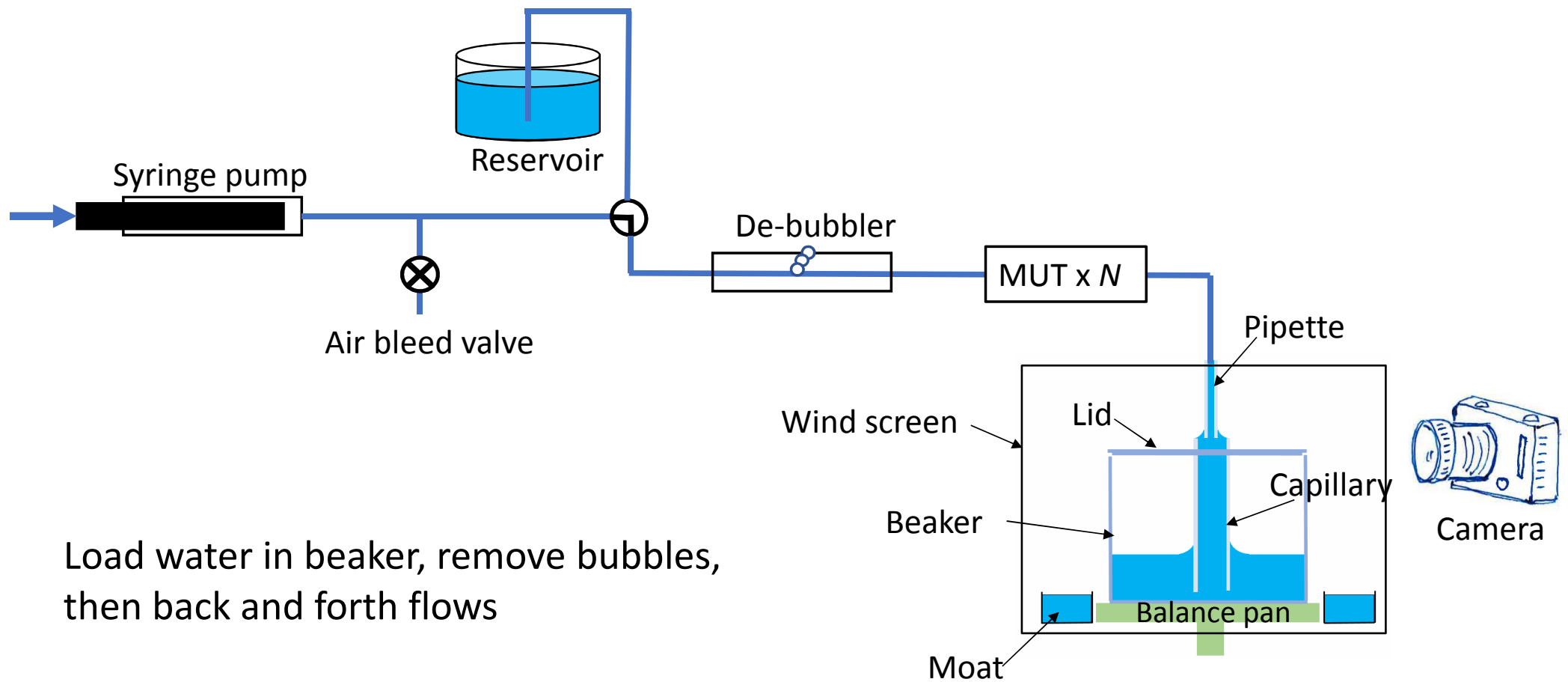


Reproducibility of Liquid Micro- Flow Measurements

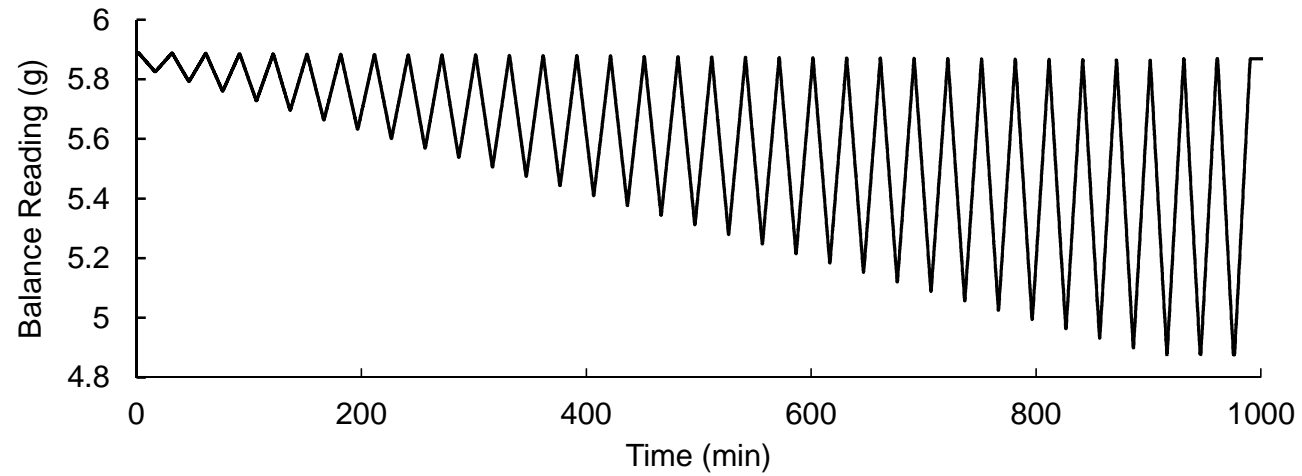
**John D. Wright and James W. Schmidt
NIST Fluid Metrology Group**

**FLOMEKO
June 26 to 28, 2019
Lisbon, Portugal**

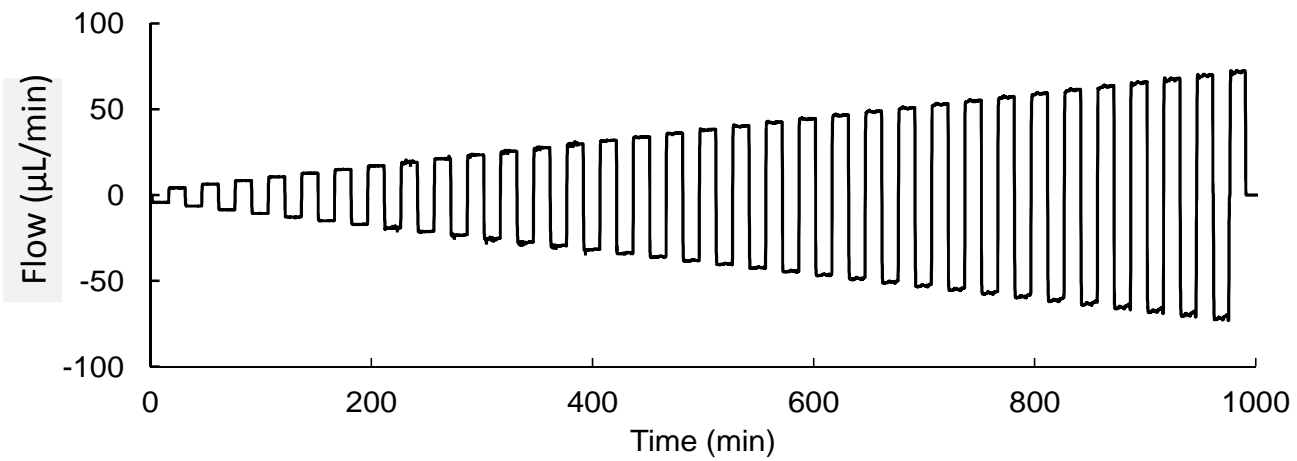
Schematic of Dynamic Gravimetric Flow Standard

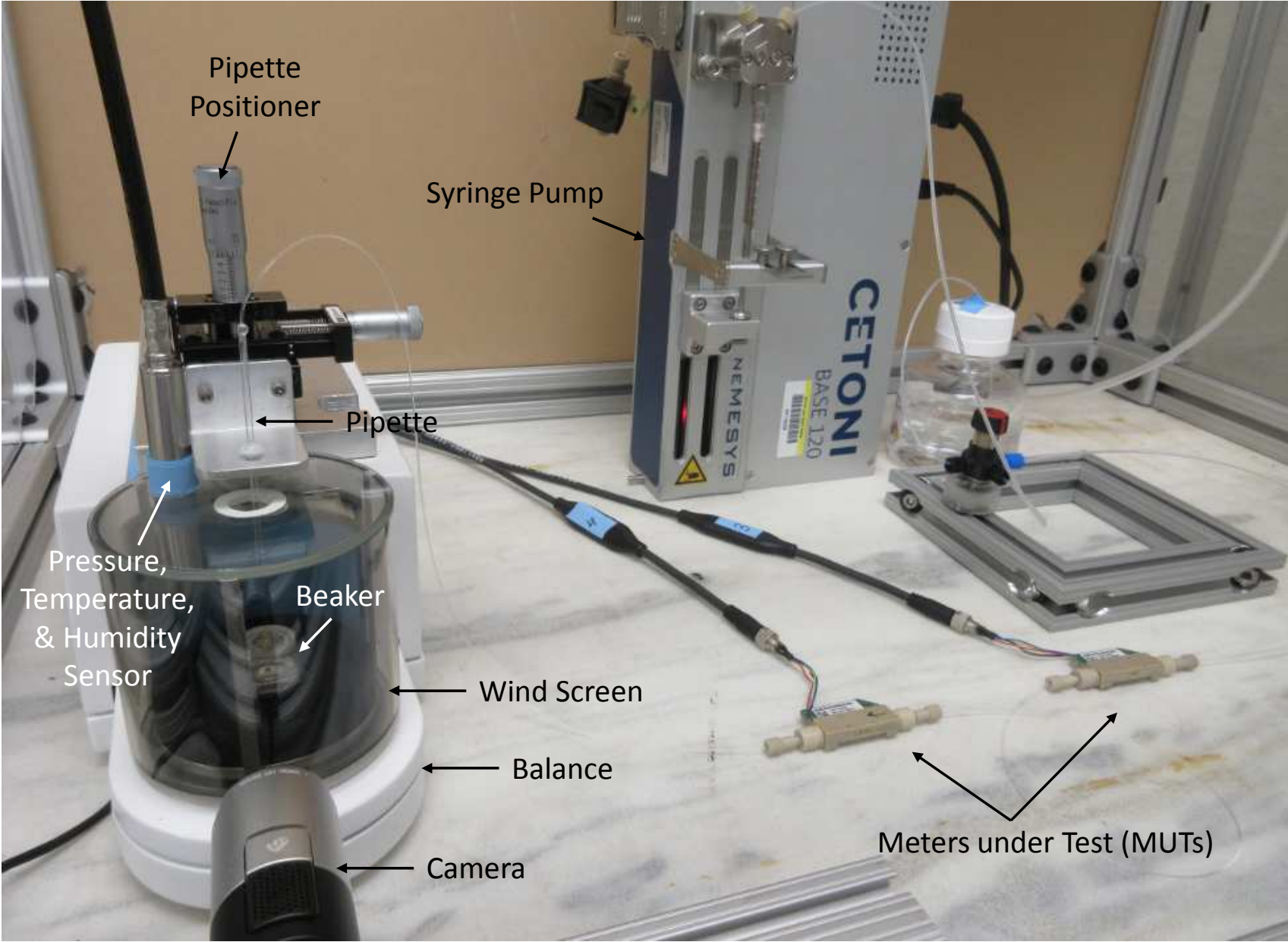


Sample Data Set: Mass and It's Time Derivative

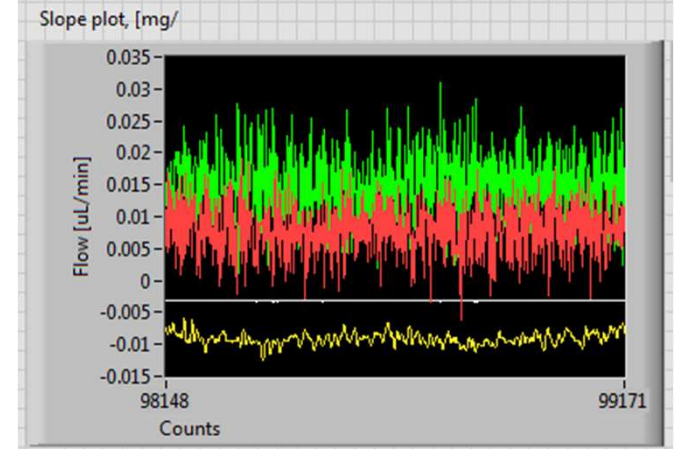
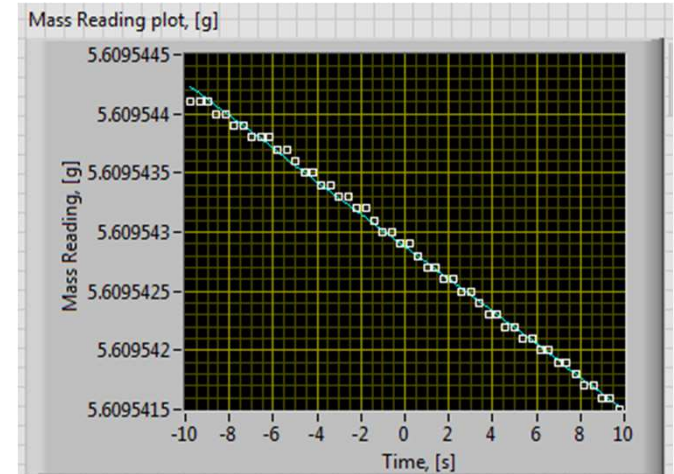
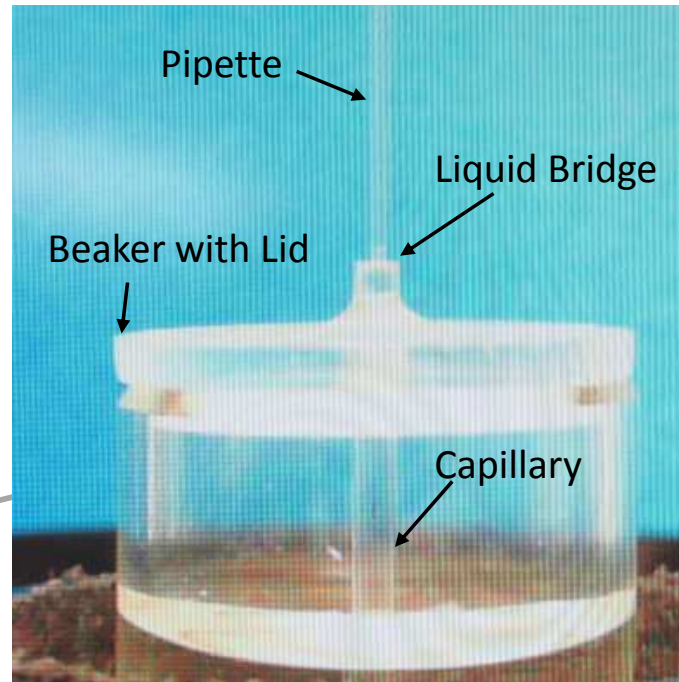
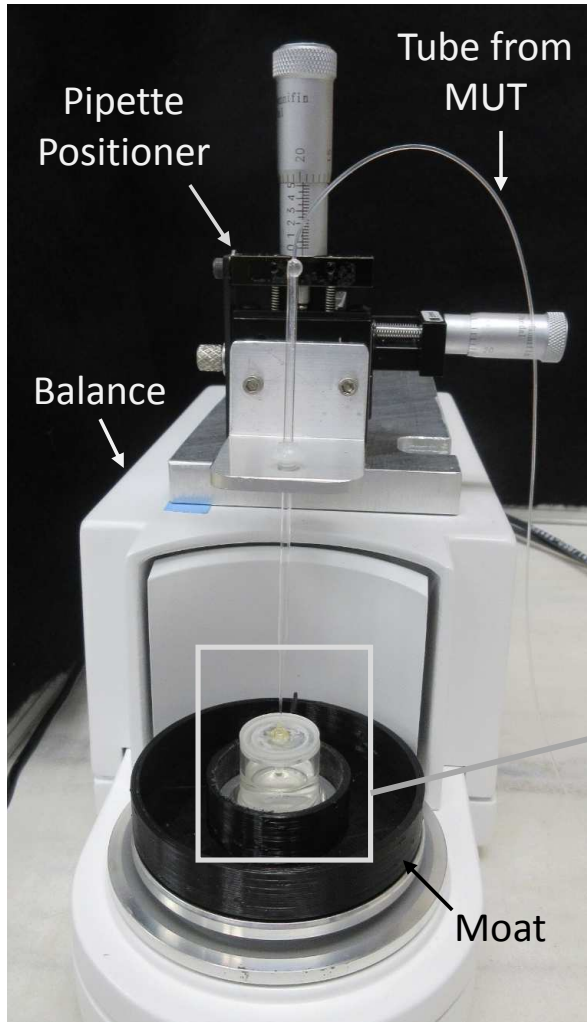


back and forth flows

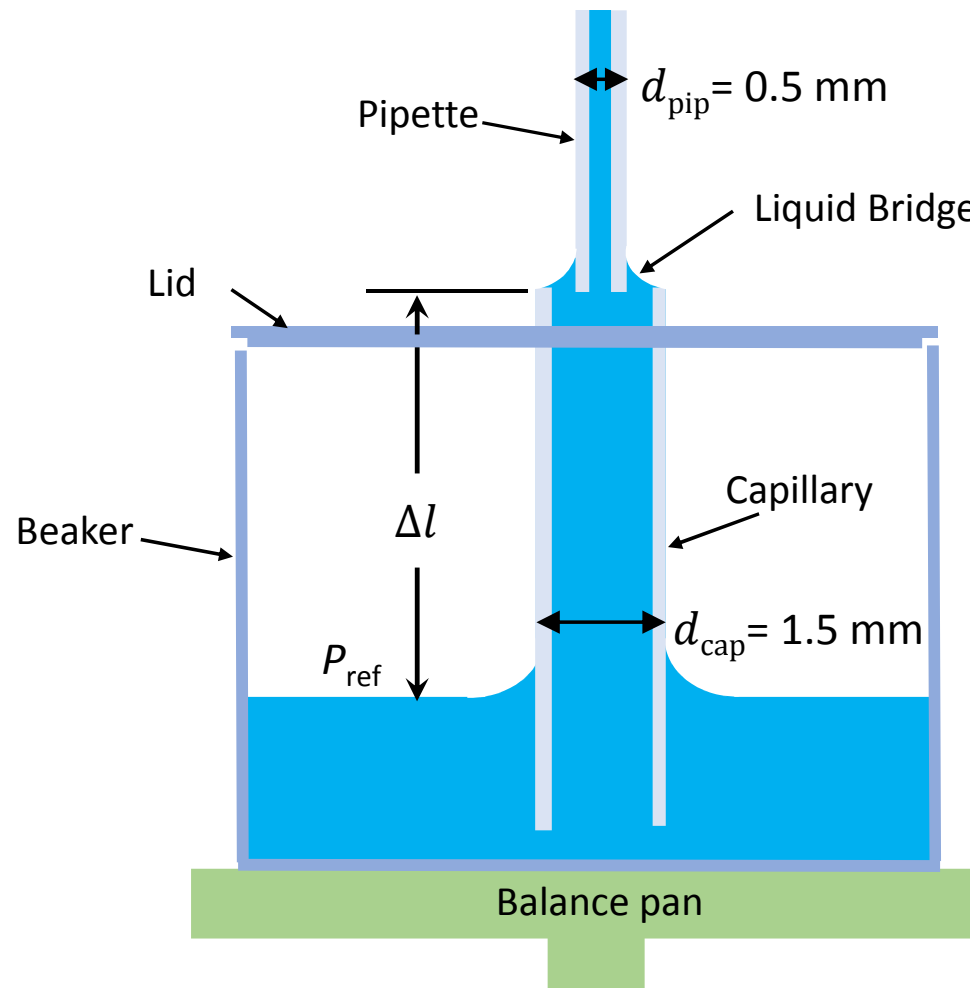




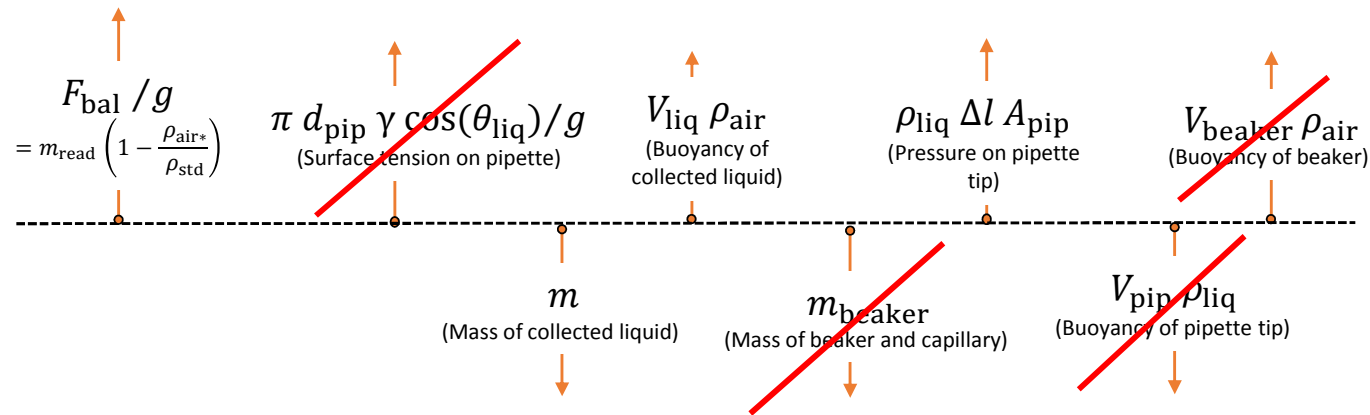
Collection Beaker on a 6 g Balance



Collection Beaker with “Liquid Bridge”



Mass Flow Equations



$$\hat{m} = m_{\text{read}} \frac{\left(1 - \frac{\rho_{\text{air}^*}}{\rho_{\text{std}}}\right)}{\left(1 - \frac{\rho_{\text{air}}}{\rho_{\text{liq}}}\right)}$$

1) Buoyancy corrections

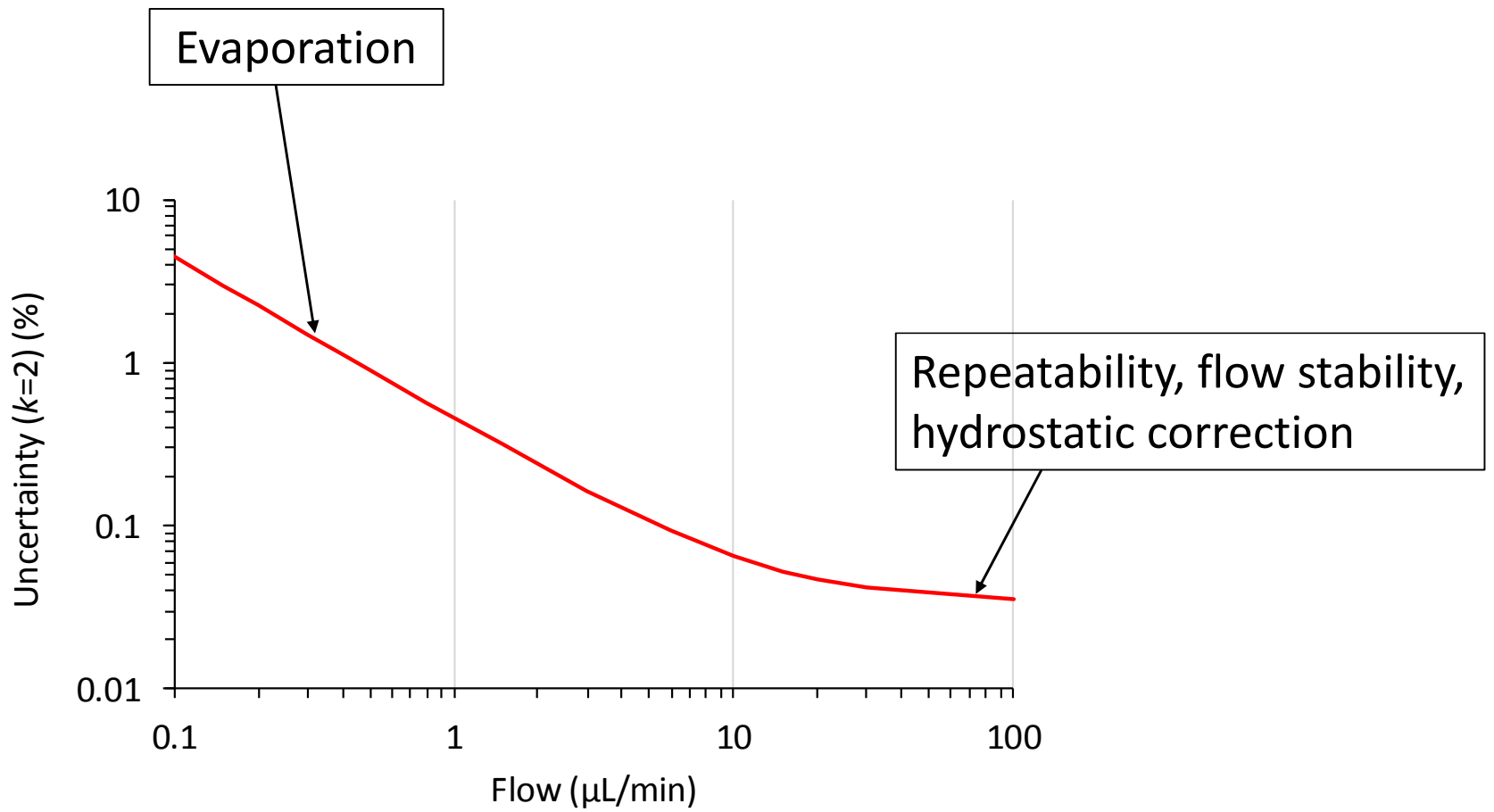
$$\dot{m}_0 = \frac{d\hat{m}}{dt} \cong \frac{N \sum_{j=1}^N t_j \hat{m}_j - \sum_{j=1}^N t_j \sum_{j=1}^N \hat{m}_j}{N \sum_{j=1}^N t_j^2 - \left(\sum_{j=1}^N t_j\right)^2}$$

2) Least squares best-fit ($N \geq 150$)

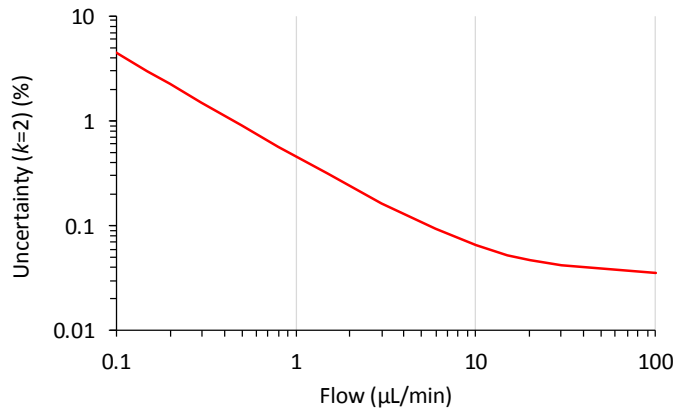
$$\dot{m} = \dot{m}_0 \left[1 - \frac{A_{\text{pip}}}{(A_{\text{beaker}} - A_{\text{cap}})}\right] + \frac{dm_{\text{evap}}}{dt}$$

3) Hydrostatic and evaporation corrections

Uncertainty Analysis



Uncertainty Analysis

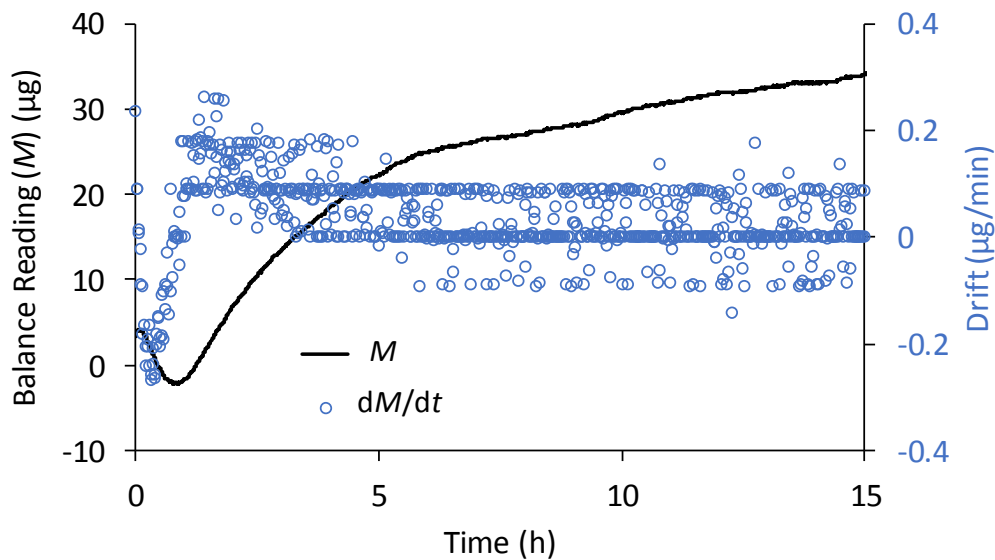


For 100 μL/min...

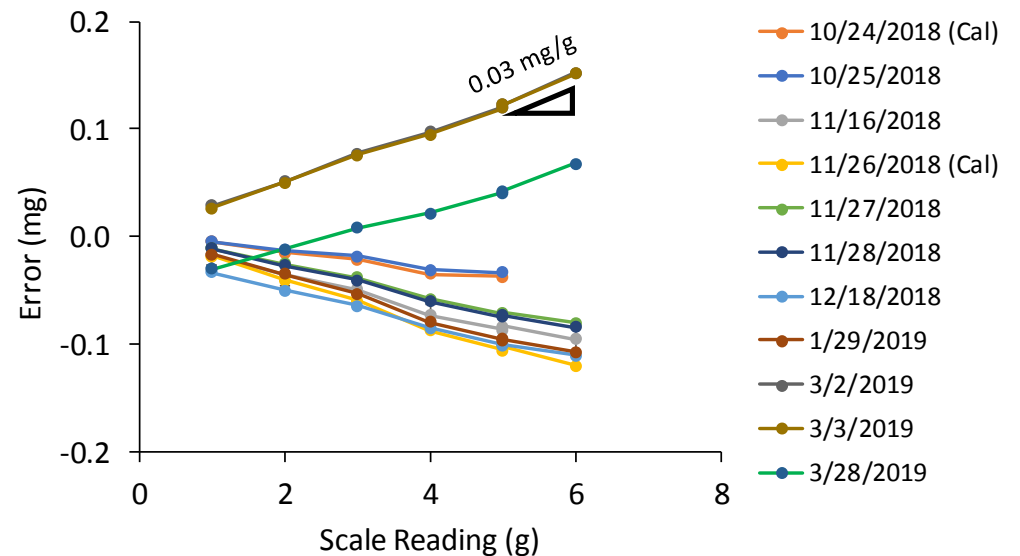
Uncertainty category	Value	Units	Standard unc. ($k = 1$)	Contrib.
			(%)	(%)
Δ Mass with buoyancy corr.	0.1	[g]	0.003	0
Δ Time	1	[min]	0.002	0
Pipette hydrostatic corr.	0.999	[-]	0.008	19
Slope calculation	0	[g/min]	0	0
Evaporation	-0.01	[μL/min]	0.002	1
Water density	0.998	[g/cm ³]	0.005	0
Repeatability & flow stability	0.1	[g/min]	0.016	79
Expanded uncertainty ($k = 2$)	0.04	[μL/min]	0.04	

Change in Mass (with Buoyancy Corrections)

At low flows: balance zero drift (0.16 $\mu\text{g}/\text{min}$)



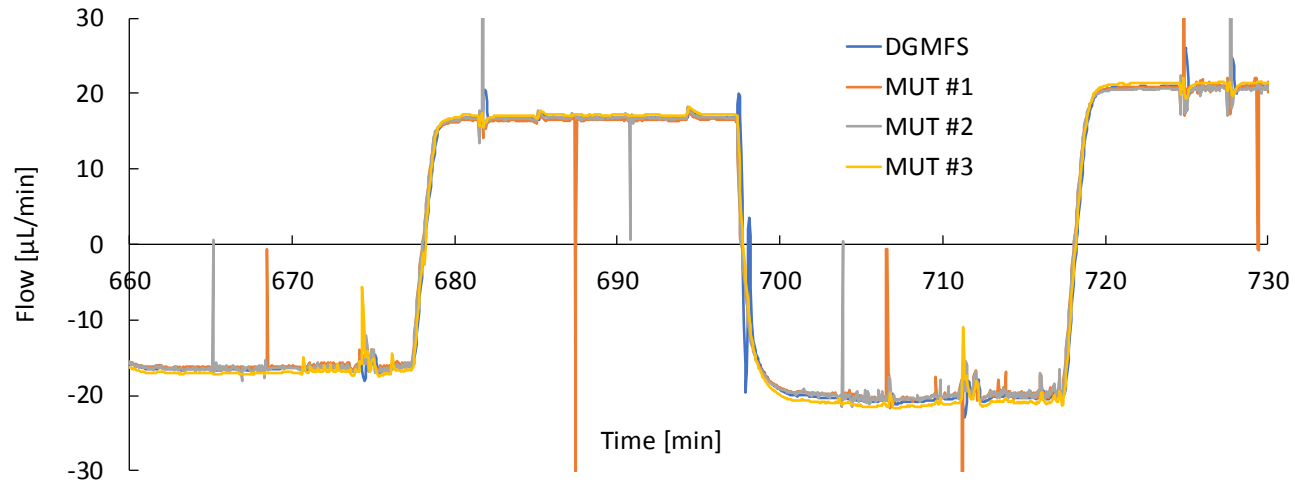
At high flows: balance calibration (0.03 mg/g)



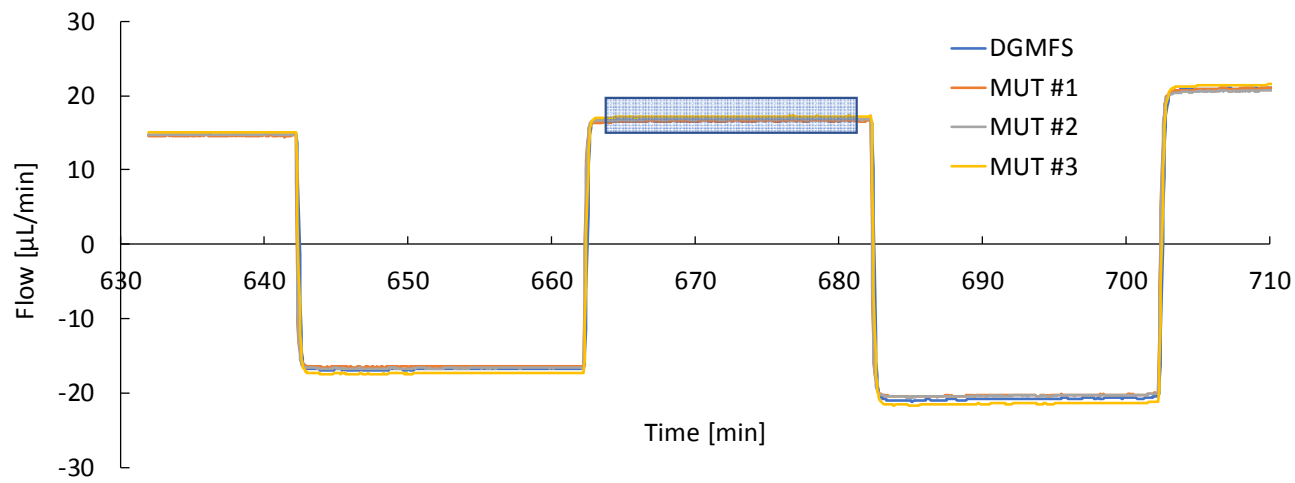
Small mass components:

- Resolution (0.1 μg) and
- Buoyancy (correction is 0.12 %, but uncertainty only 5 PPM)

Flow Stability

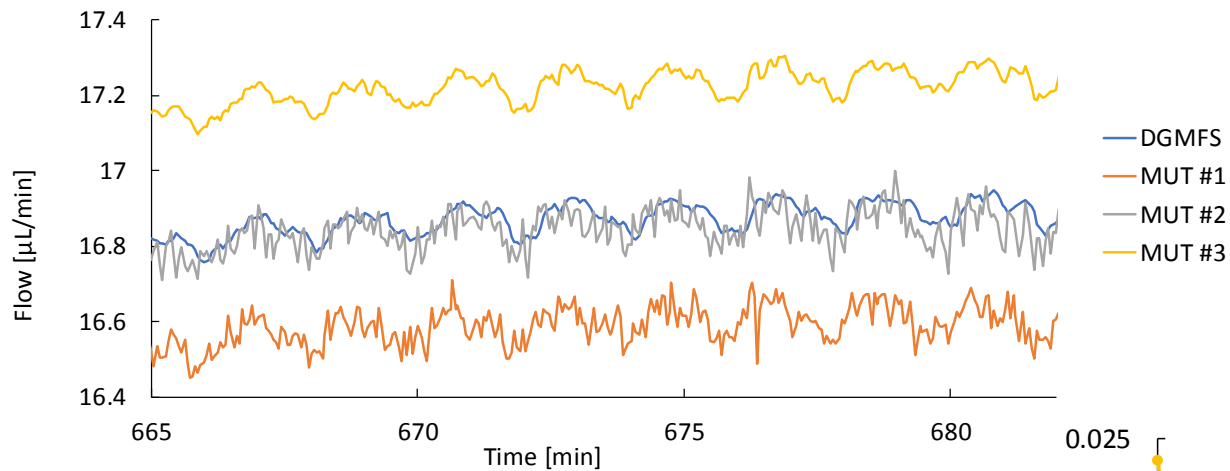


Air bubbles present

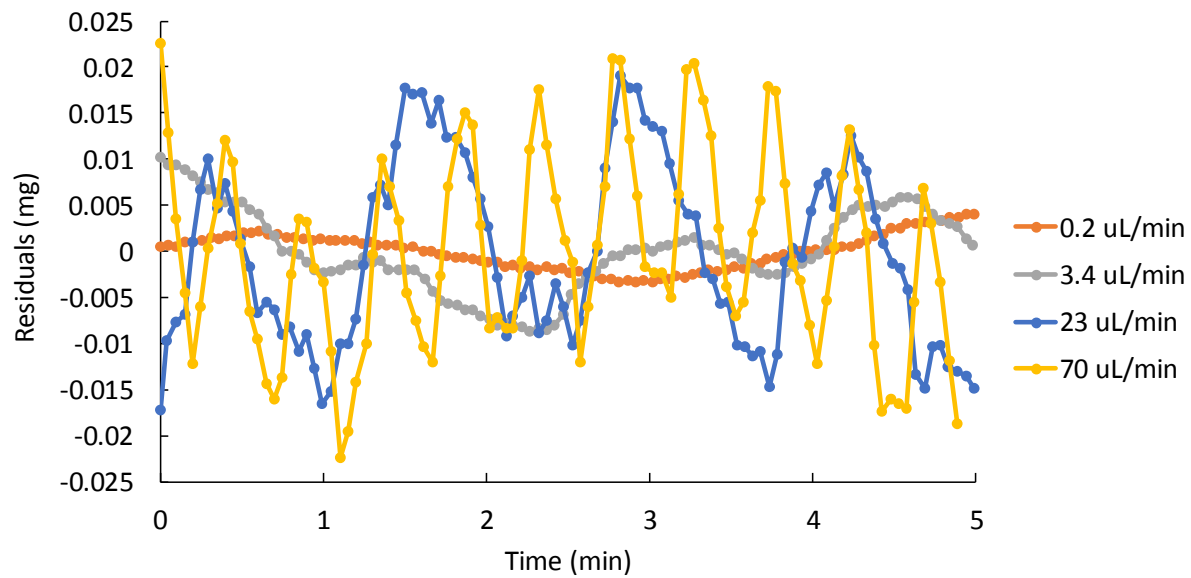


No air bubbles

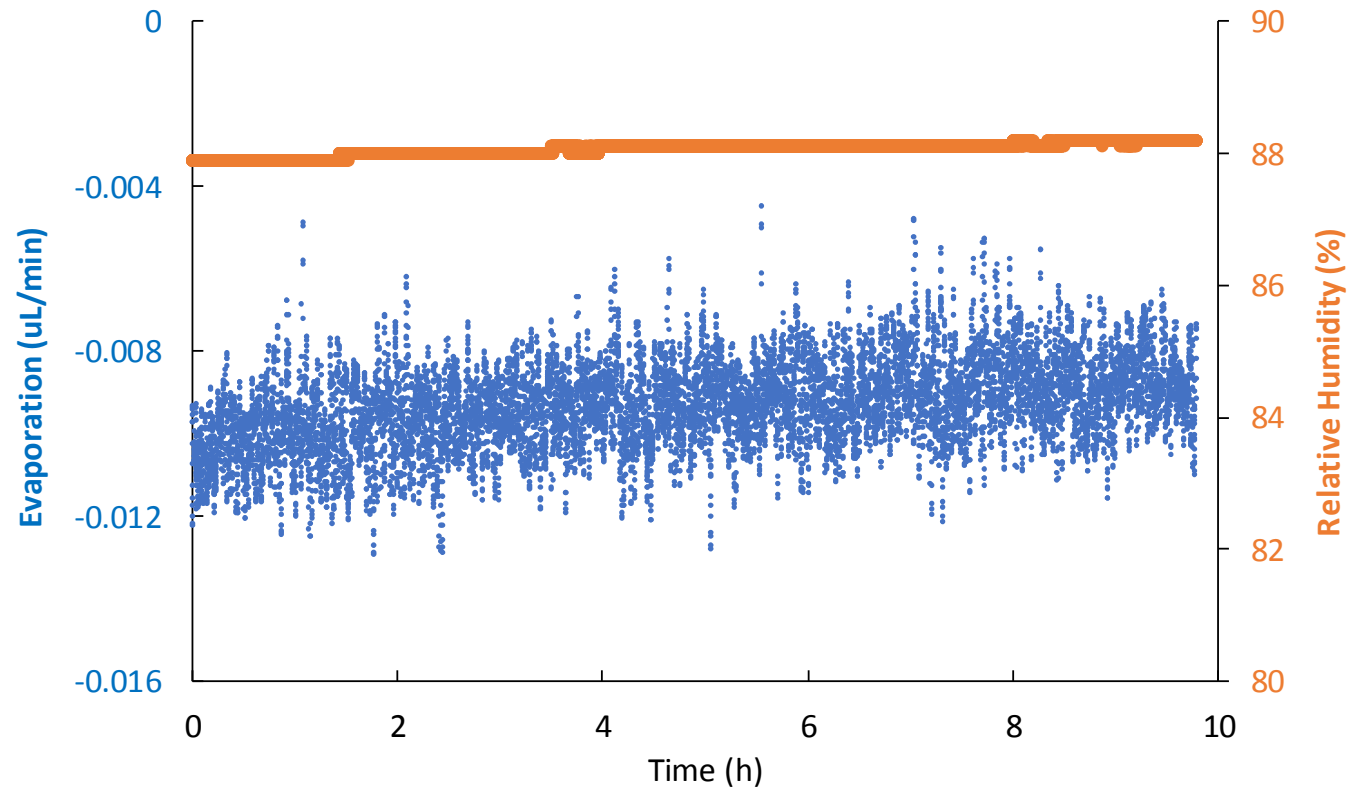
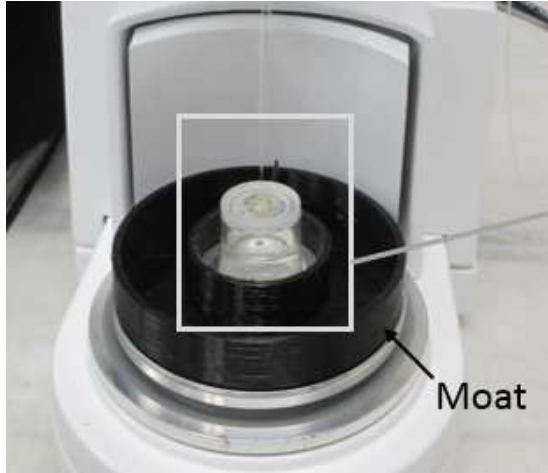
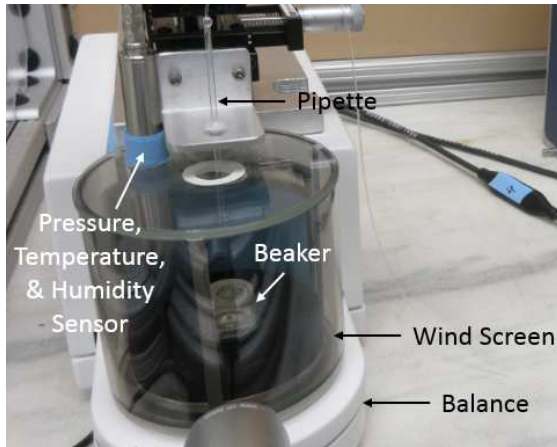
Flow Stability



Type A repeatability:
standard deviation of 5 averages

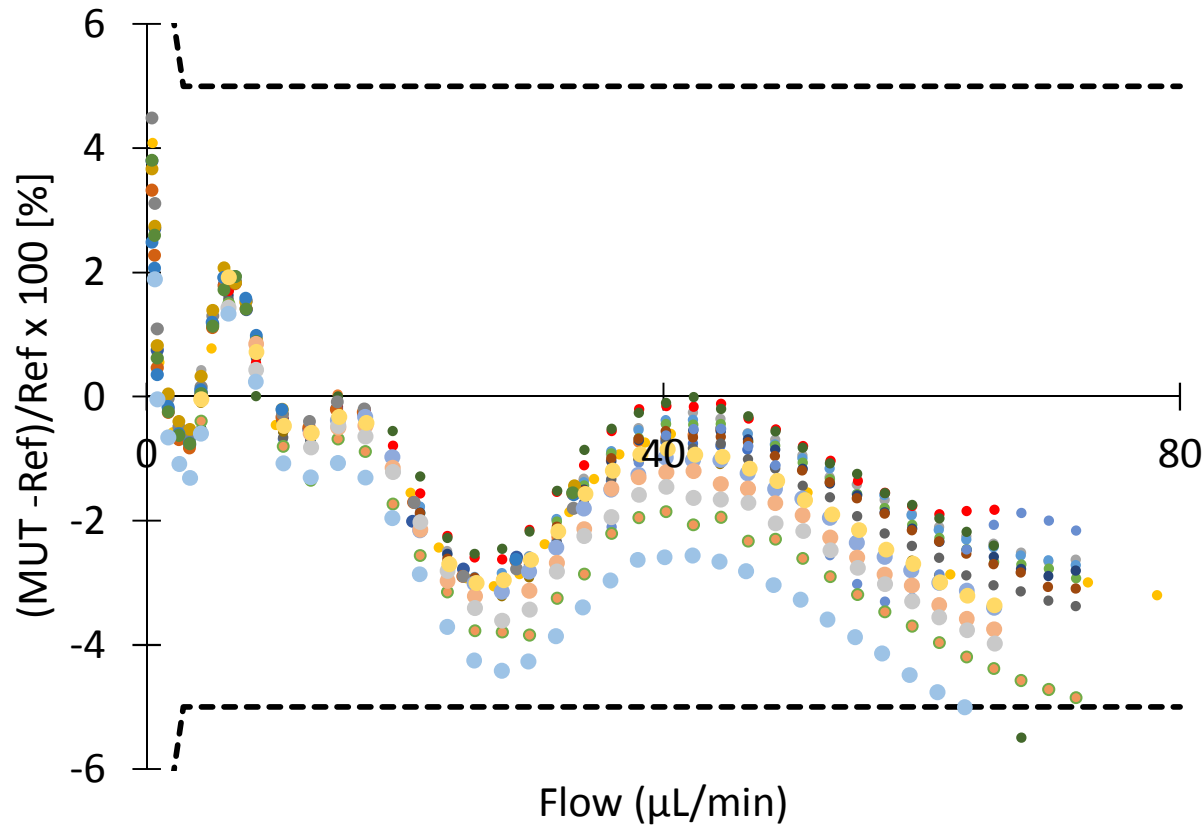


Evaporation Correction

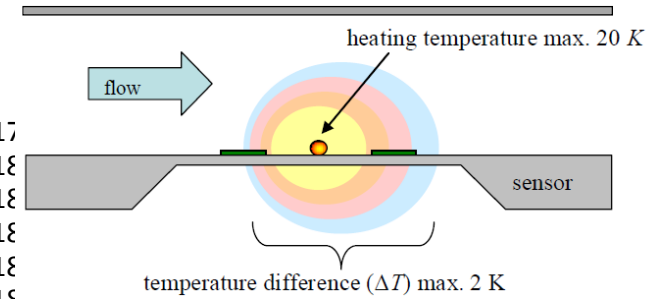


Correction $\sim -0.01 \mu\text{L}/\text{min}$, standard uncertainty = $0.002 \mu\text{L}/\text{min}$

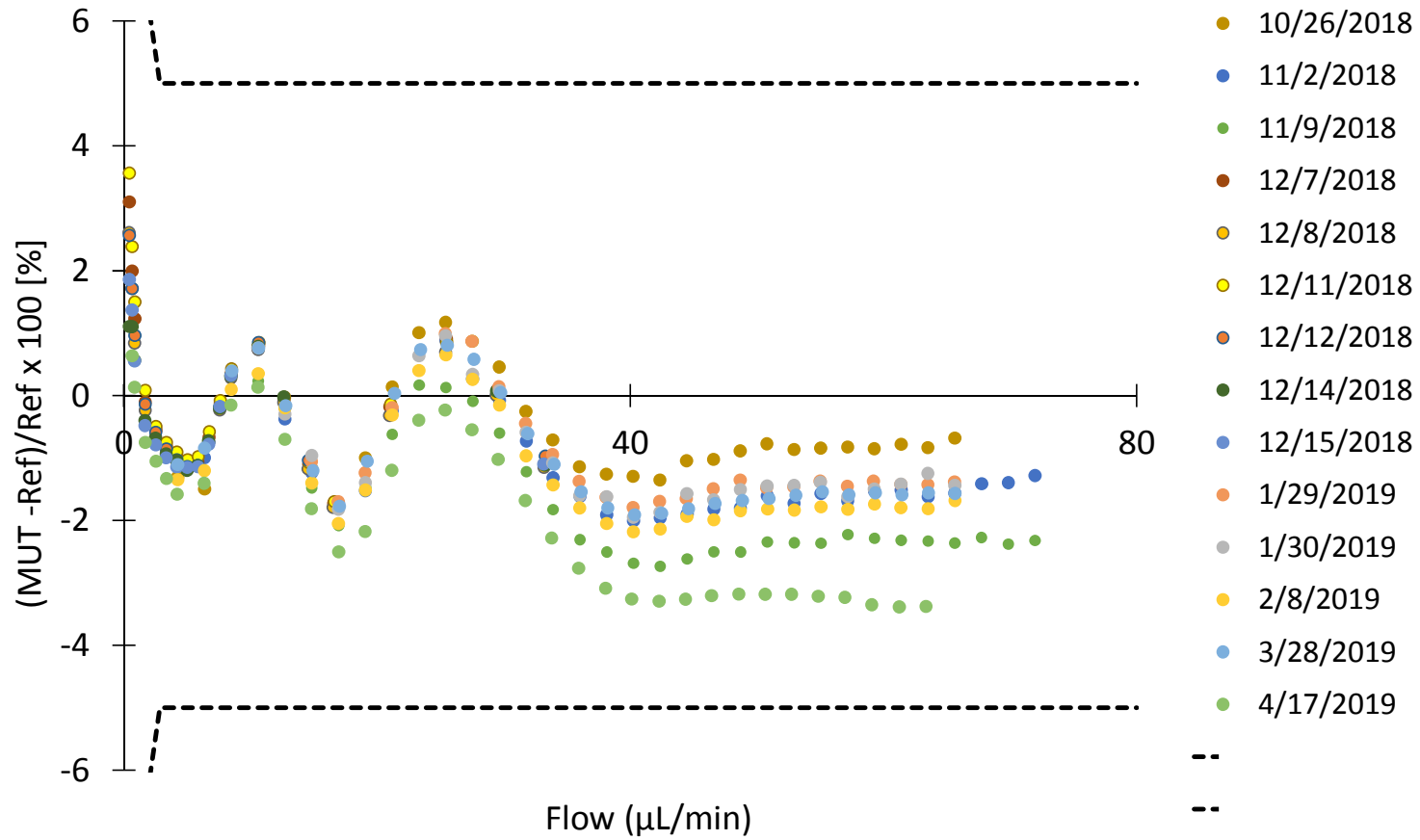
Thermal Meter A



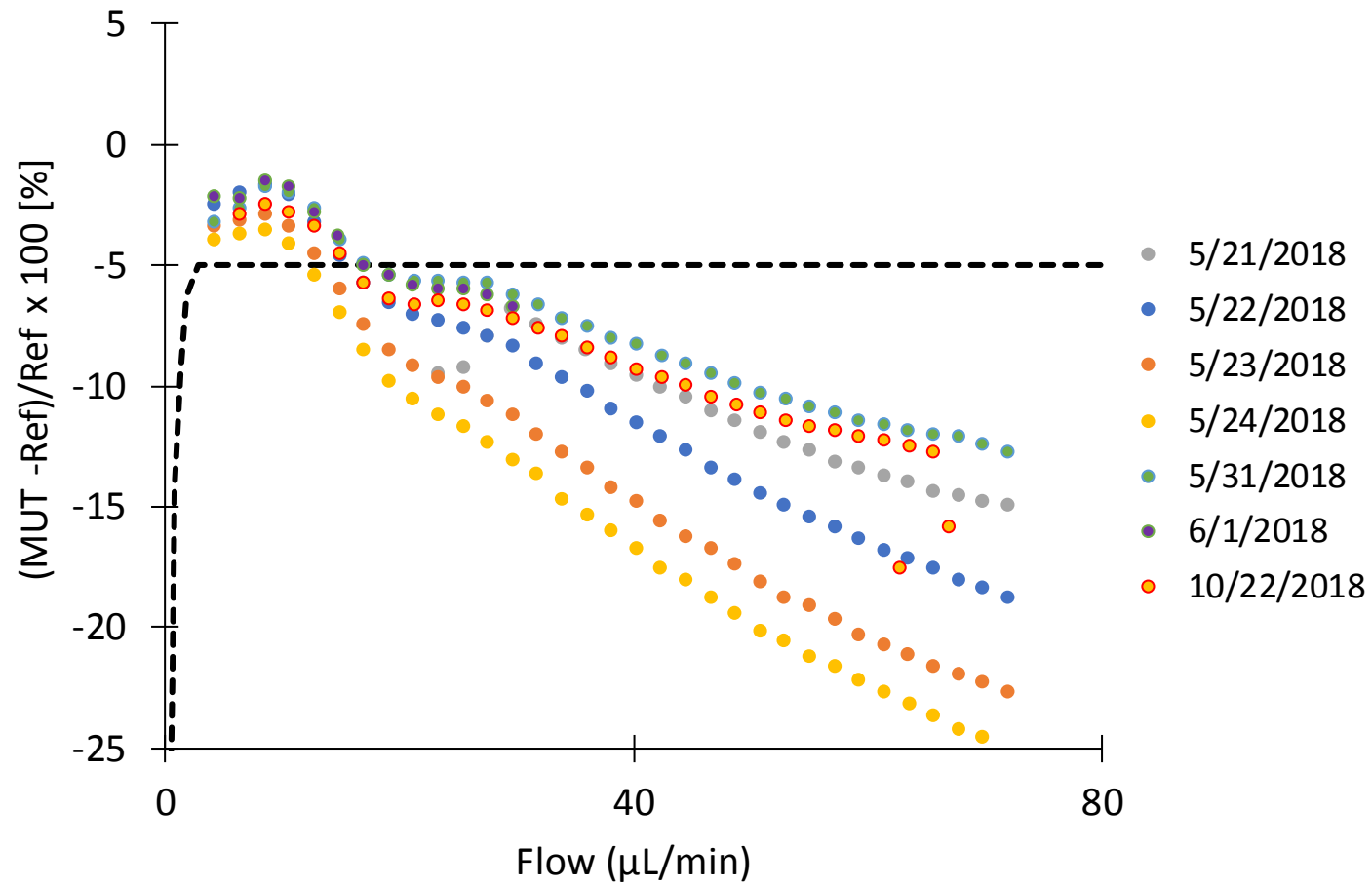
- 7/18/2017
- 5/10/2018
- 5/11/2018
- 5/16/2018
- 5/18/2018
- 5/19/2018
- 5/20/2018
- 5/22/2018
- 5/23/2018
- 5/24/2018
- 10/22/2018
- 10/25/2018
- 11/2/2018
- 11/9/2018
- 12/7/2018
- 12/8/2018
- 12/11/2018
- 12/12/2018
- 12/14/2018
- 12/15/2018
- 1/29/2019
- 1/30/2019
- 2/8/2019
- 3/28/2019
- 4/17/2019



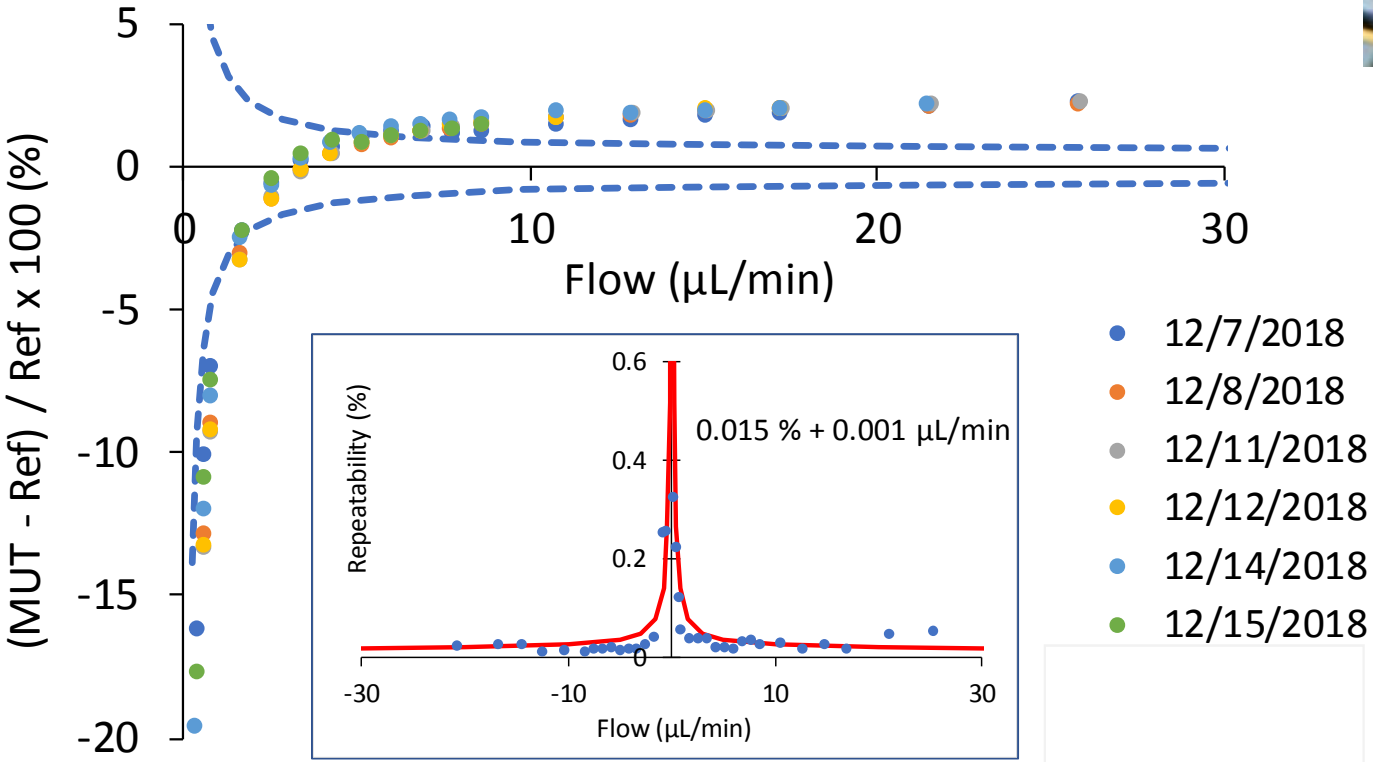
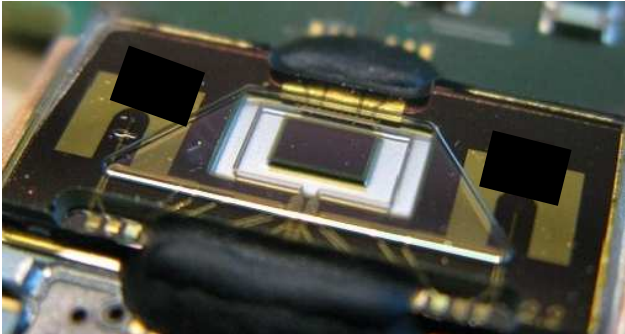
Thermal Meter B



Thermal Meter C



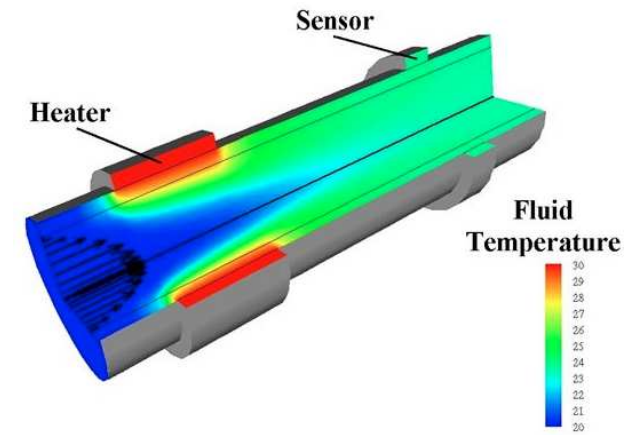
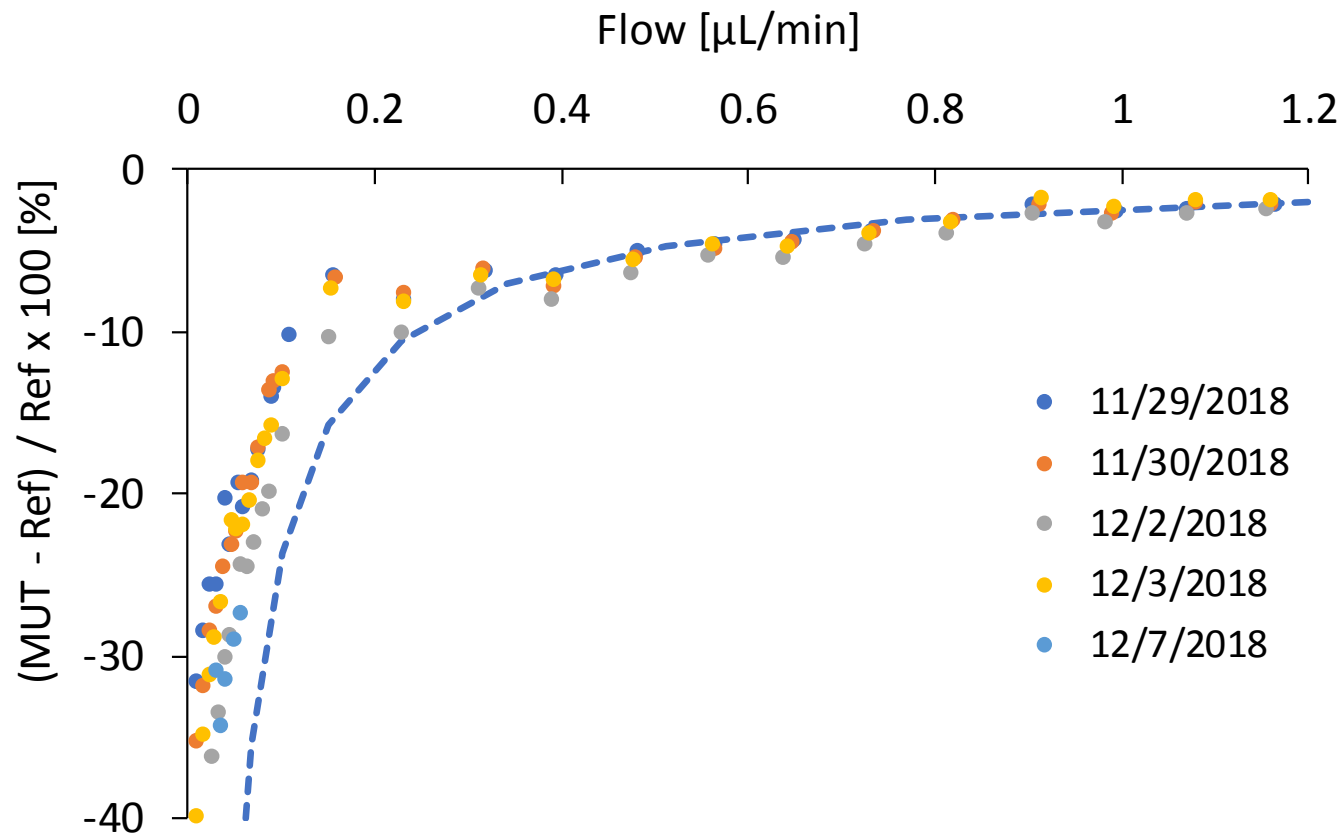
Coriolis Meter D



Standard deviation of 5 one-minute averages

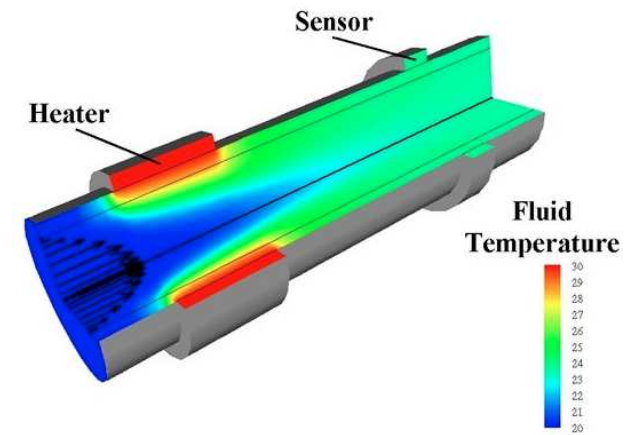
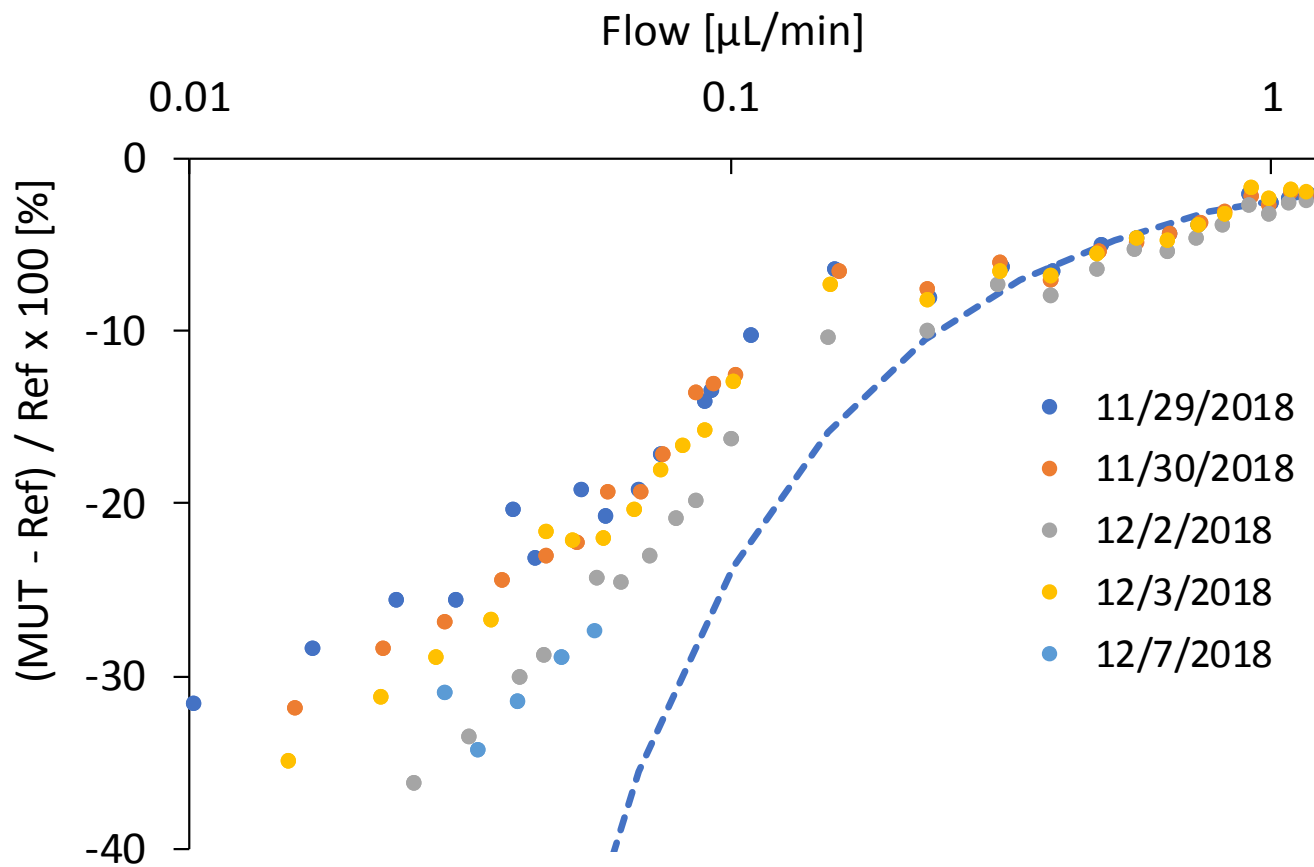
NOT re-zeroed

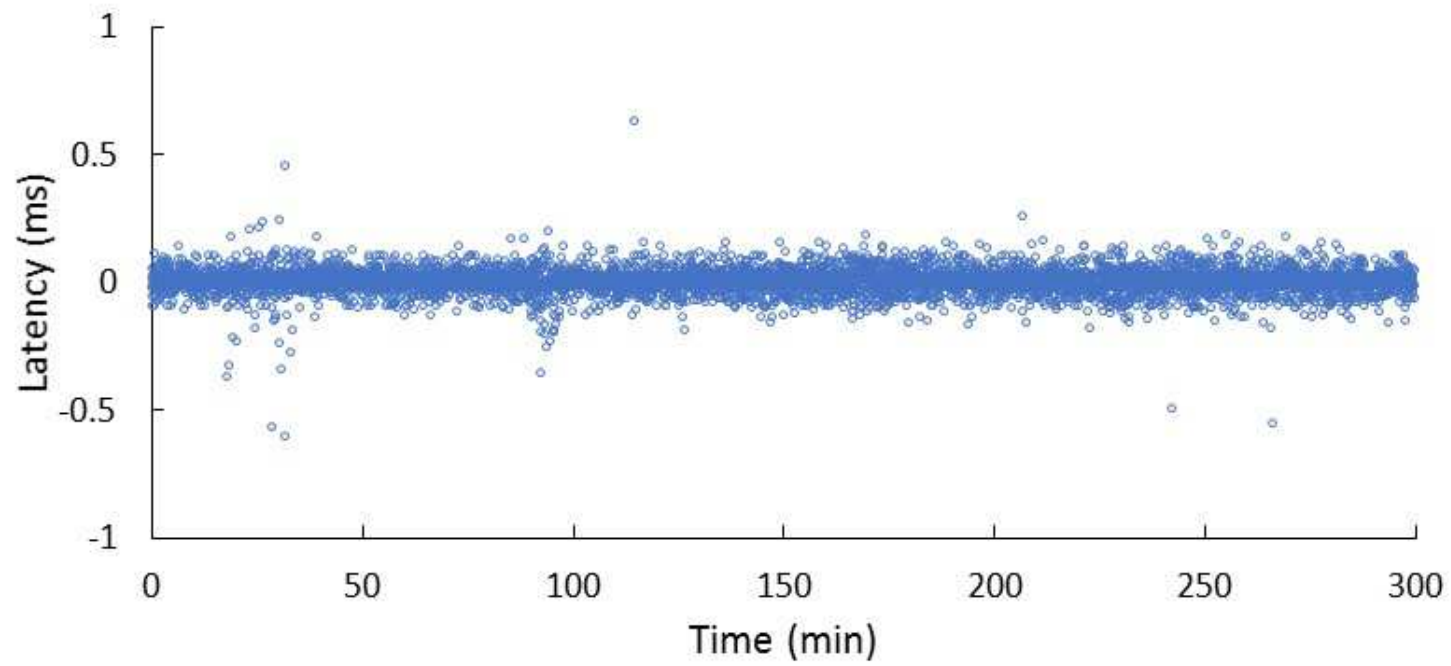
Thermal Meter E



Summary and Conclusions

- Improvements: liquid bridge, evaporation trap, camera, pipette positioner
- Derived flow equations and presented uncertainty analysis
- At high flows: repeatability and hydrostatic corrections
- At low flows: evaporation corrections and repeatability
- Calibrations of commercial meters: stable within 1.5 % for many months
- Flow meter uncertainty in user's application can be significantly improved (3x) by calibration against a reference flow standard





Log7262018.xlsx

