

Efficacy of FTIR Analysis in Determining CO₂ Loading on Diglycolamine

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Outline

- Liquid amine background
- Microgravity adaptation
- FTIR motivation
- Experimental Method
 - pH Desorption
 - FTIR Analysis/ Calibration
- Results
- Discussion



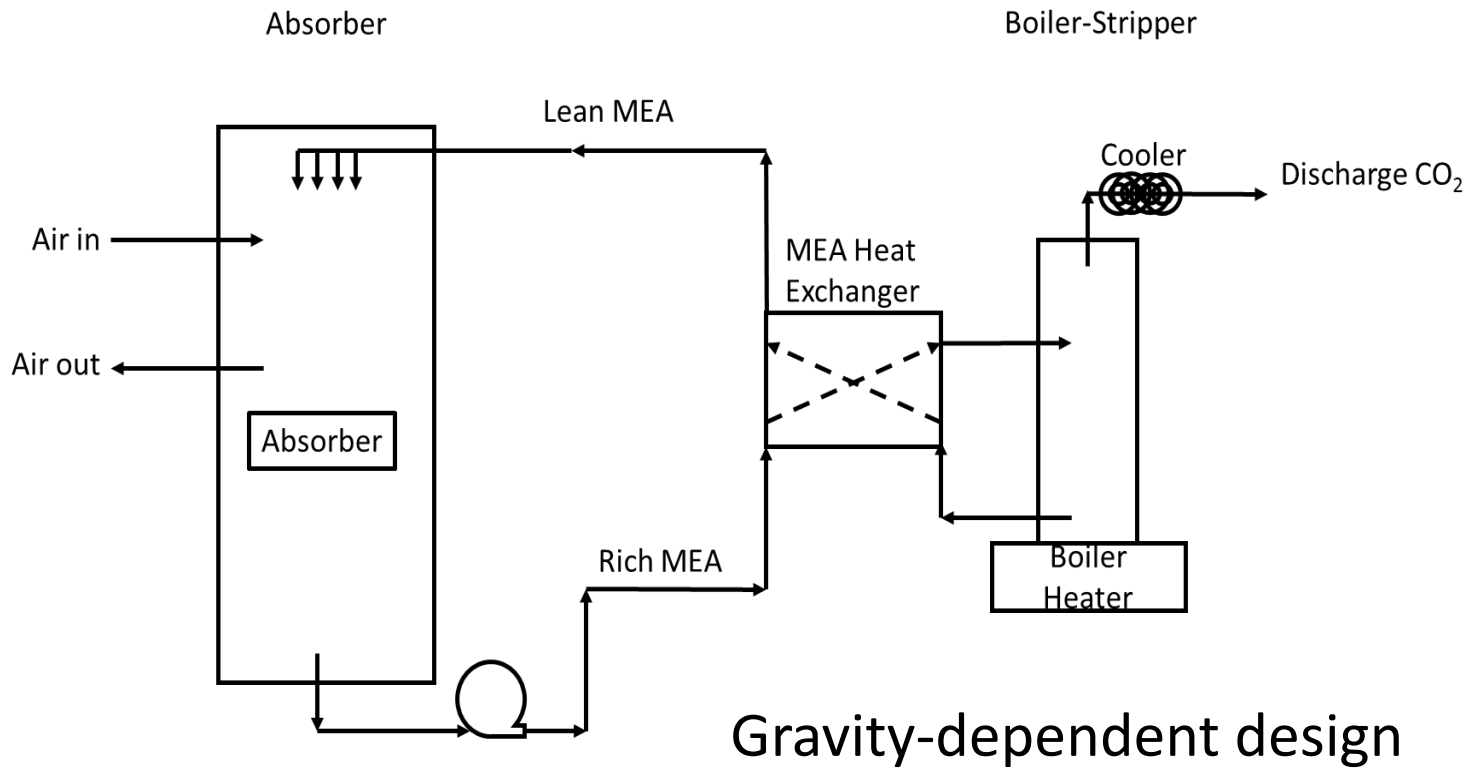
Liquid Amine Background

- Industrial plant gas sweetening (H_2S , CO_2 removal)
- Liquid amine systems used in submarines since the 1950s
 - Monoethanolamine (MEA) scrubbers
 - Maintained CO_2 concentration at 1% of the atmosphere
- Advantages over solid sorbent bed systems
 - Power and volume savings
- Potential usage in human spaceflight
 - JSC evaluation of various liquid amines in FY16



Liquid Amine Background

Typical CO₂ removal plant process diagram



Microgravity Adaptation

Goal: design a liquid amine-based CO₂ capture system that can operate in microgravity

- Contactor designs for adsorbing and desorbing
- Captured liquid flow
- CO₂ mass flux
- Separation of gas and liquid



Microgravity Adaptation

Adsorb vs Degas (regeneration) mechanics for Diglycolamine (DGA)

- Tasks split between JSC and ARC

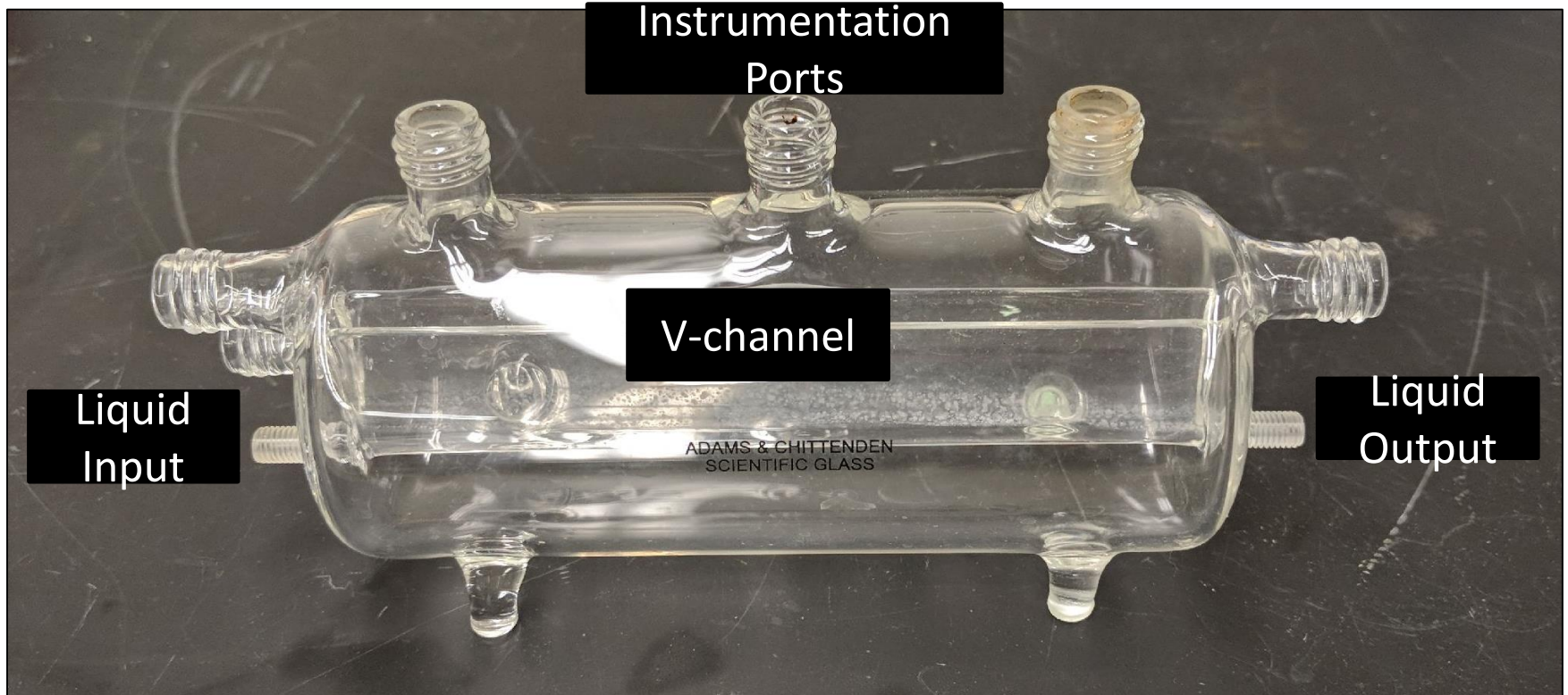
Focal points for ARC:

- Degas rate
 - Temperature
 - Surface Area
 - Liquid Flowrate (contact time)
 - CO₂ loading
 - H₂O concentration



Degasser Design

Goal: Design a scaled-down v-channel contactor that facilitates characterization of DGA degas mechanics

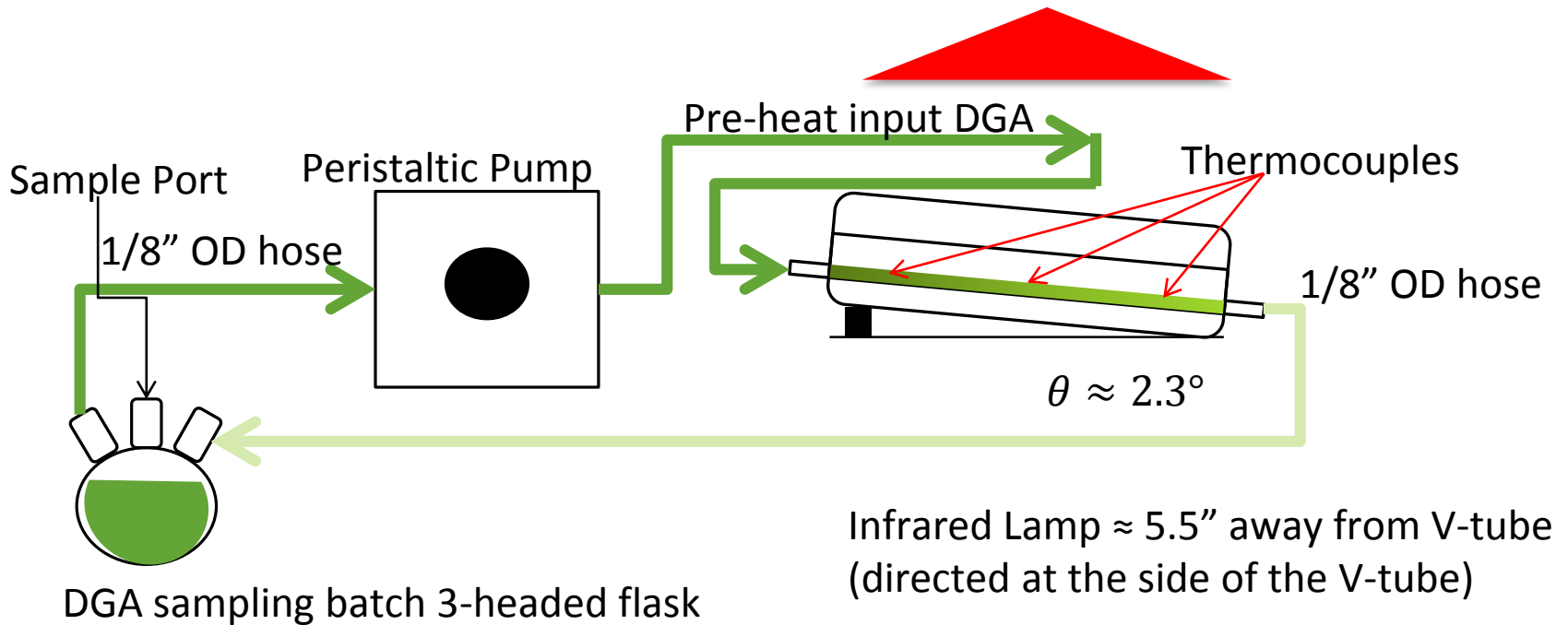


Glass V-tube



Degasser Design

Continuous flow sampling



FTIR Motivation

How do we measure CO₂ loading in the DGA?

- ~~Solution pH level~~
- ~~Viscosity~~
- ~~Raman Spectroscopy~~
- Fourier Transform Infrared Spectroscopy (FTIR)
 - MEA CO₂ loading (Einbu, Aslak, et al., 2012)
- pH desorption method (Rogers, Tanya, et al., 2017)
 - “Acid desorption” “Acid Test”
 - 96%-98% CO₂ recovery (Zhou, Shan, et al., 2010)

pH desorption requires ~1mL of solution

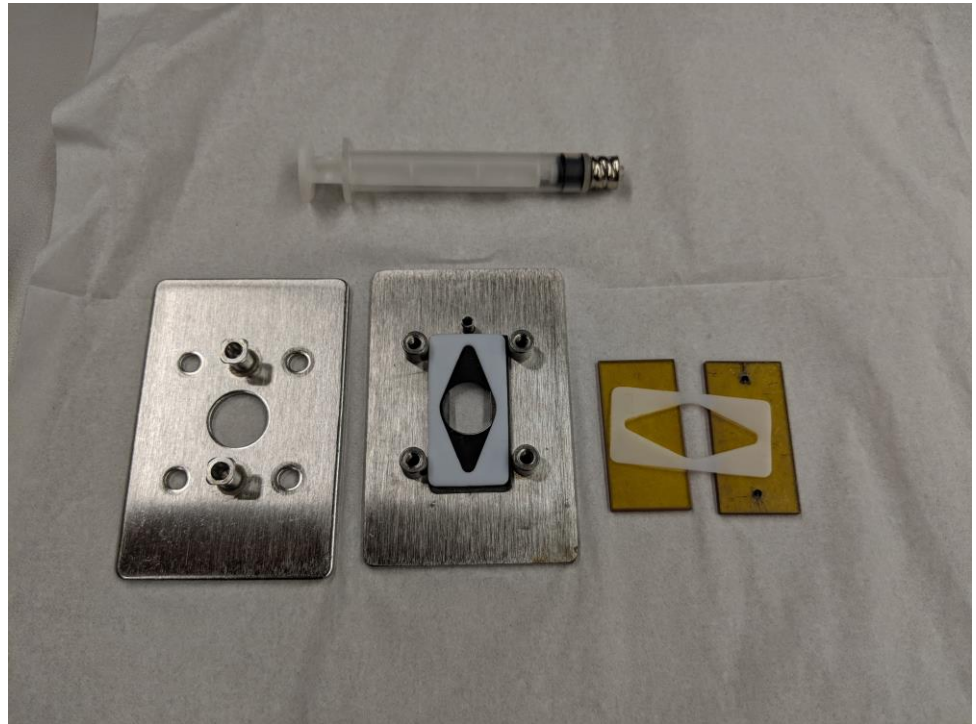
FTIR can potentially only require ~100μL



FTIR Procedure

Buck Scientific demountable IR window liquid cell

- 4mm ZnSe crystal windows
- 0.015mm sample spacer
- Samples loaded with luer lock gastight syringe
- Entire window disassembled and cleaned with DI water and isopropanol between sample analysis runs



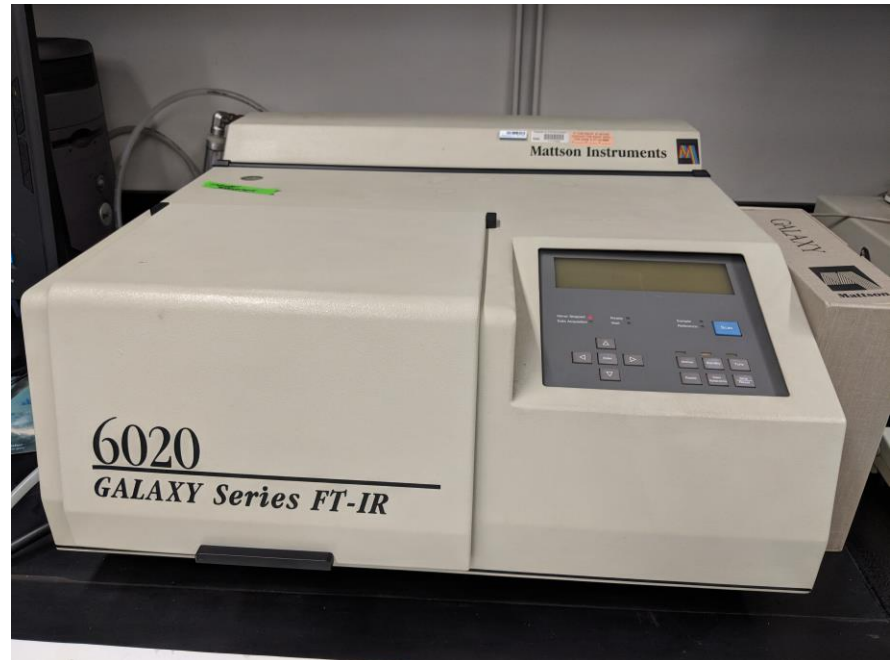
FTIR Procedure

Mattson Galaxy 6020 FTIR using WinFIRST v2.10

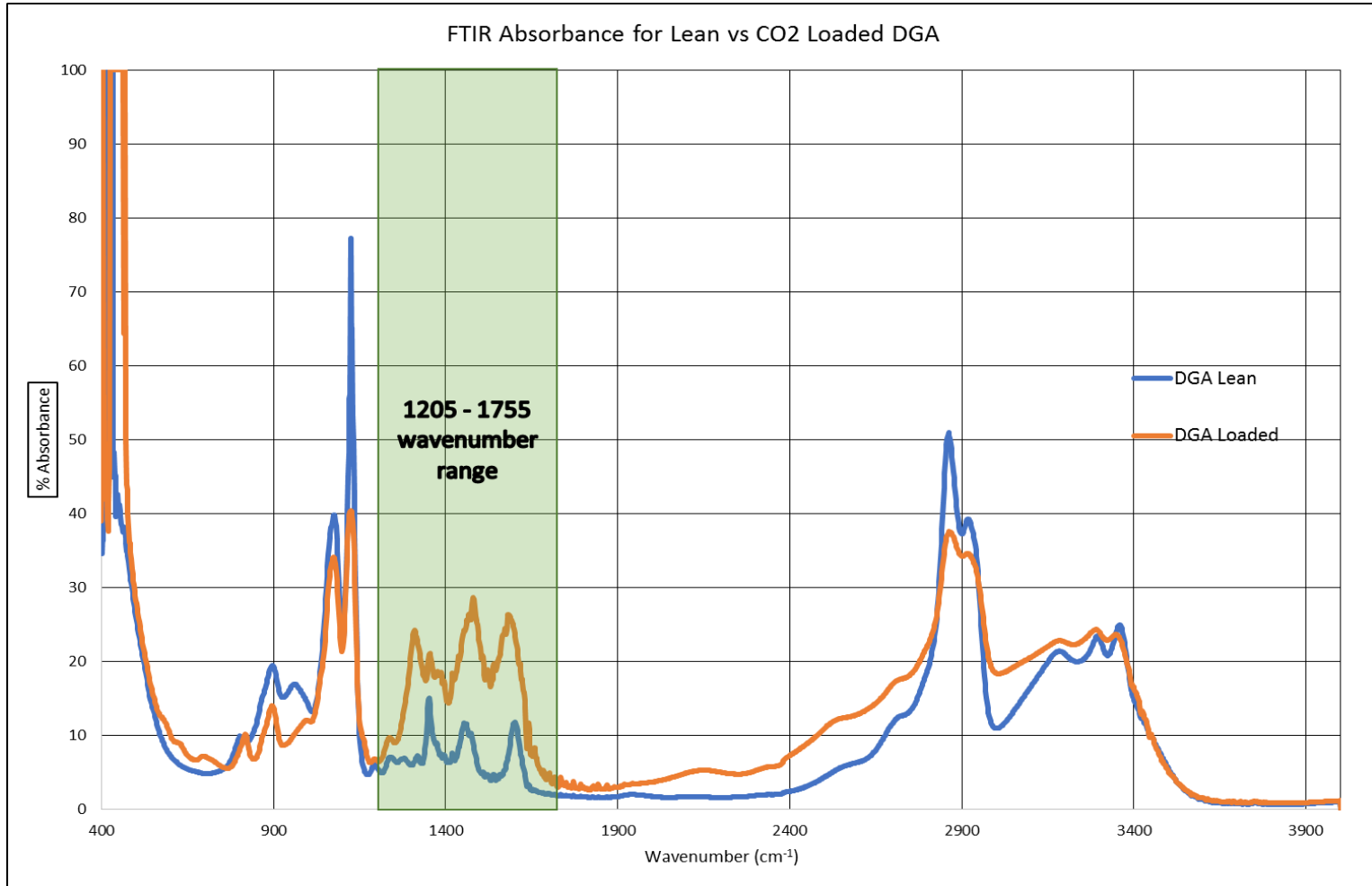
- Transmittance collected at 10kHz
- Wavenumbers 400 – 4000 cm^{-1}
- Resolution = 4

Spectragryph V1.1.0

- Baselines and pre-loaded calibration standards
- Experimental samples normalized on peak at 894 cm^{-1}
 - Peak that remained consistent in all samples of DGA

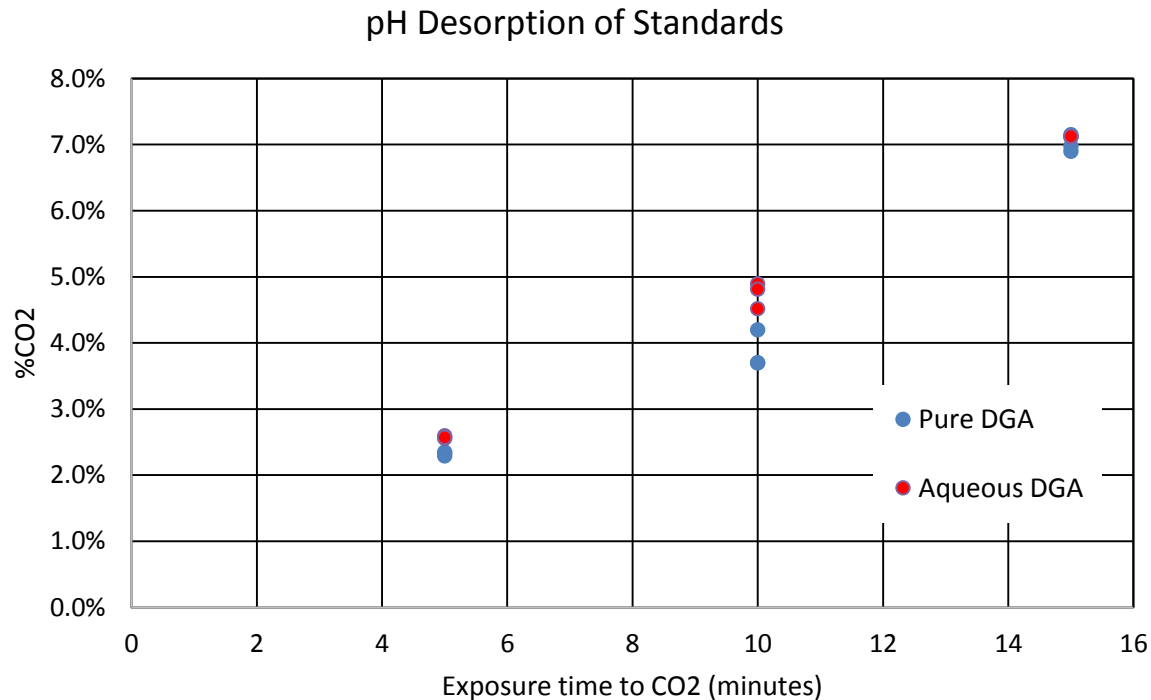


FTIR Calibration

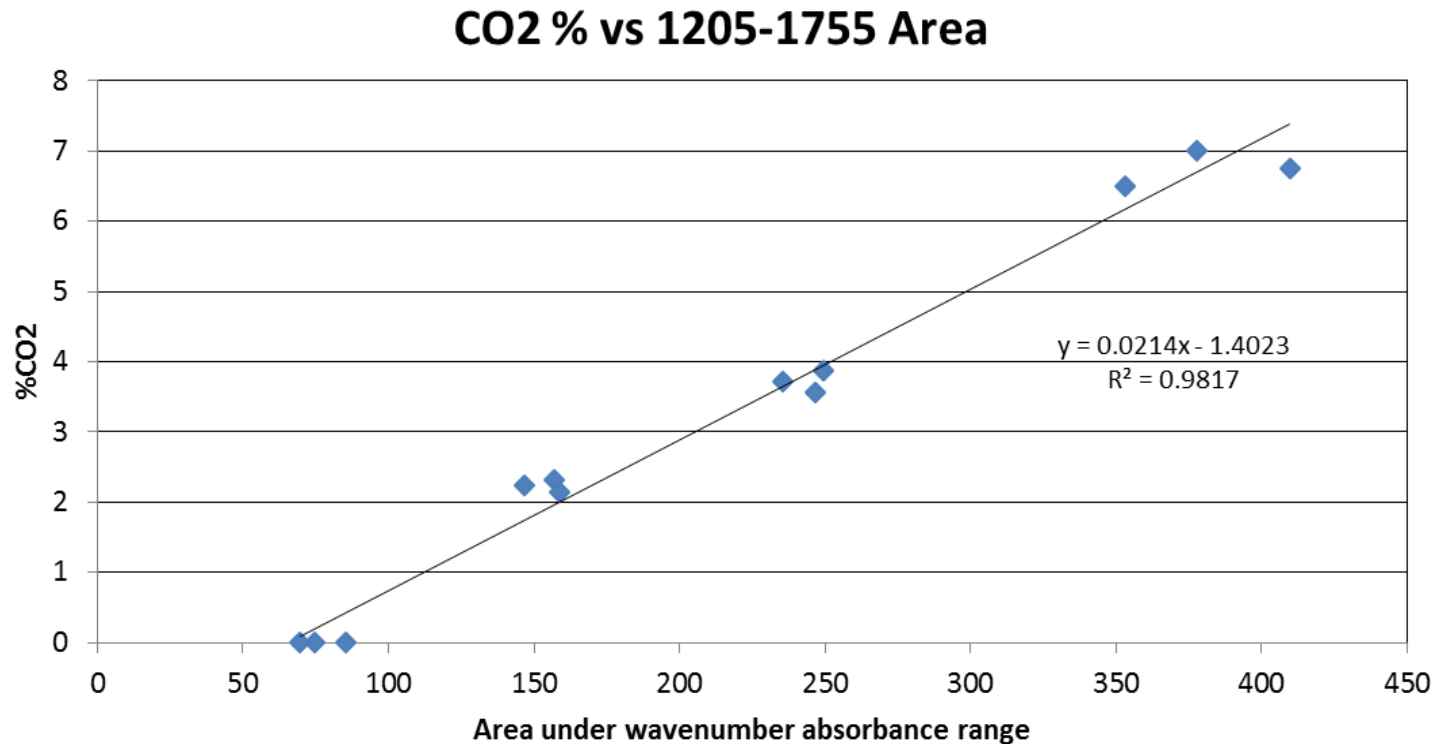


FTIR Calibration

Calibrate against pH desorption for range of CO₂ % loading on DGA



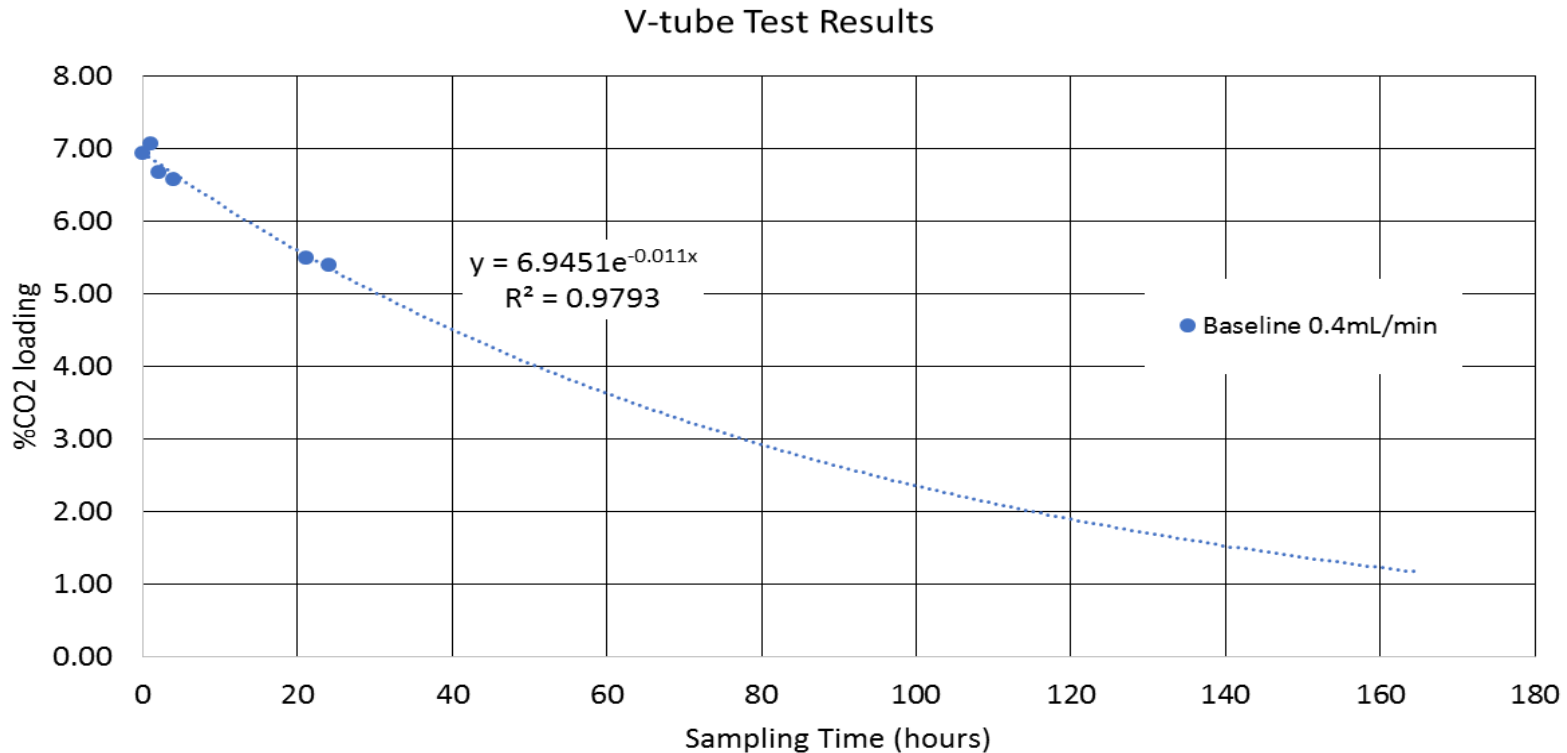
FTIR Calibration



Calibration points for DGA run at various concentrations of water solution (0%, 4%, and 8%)



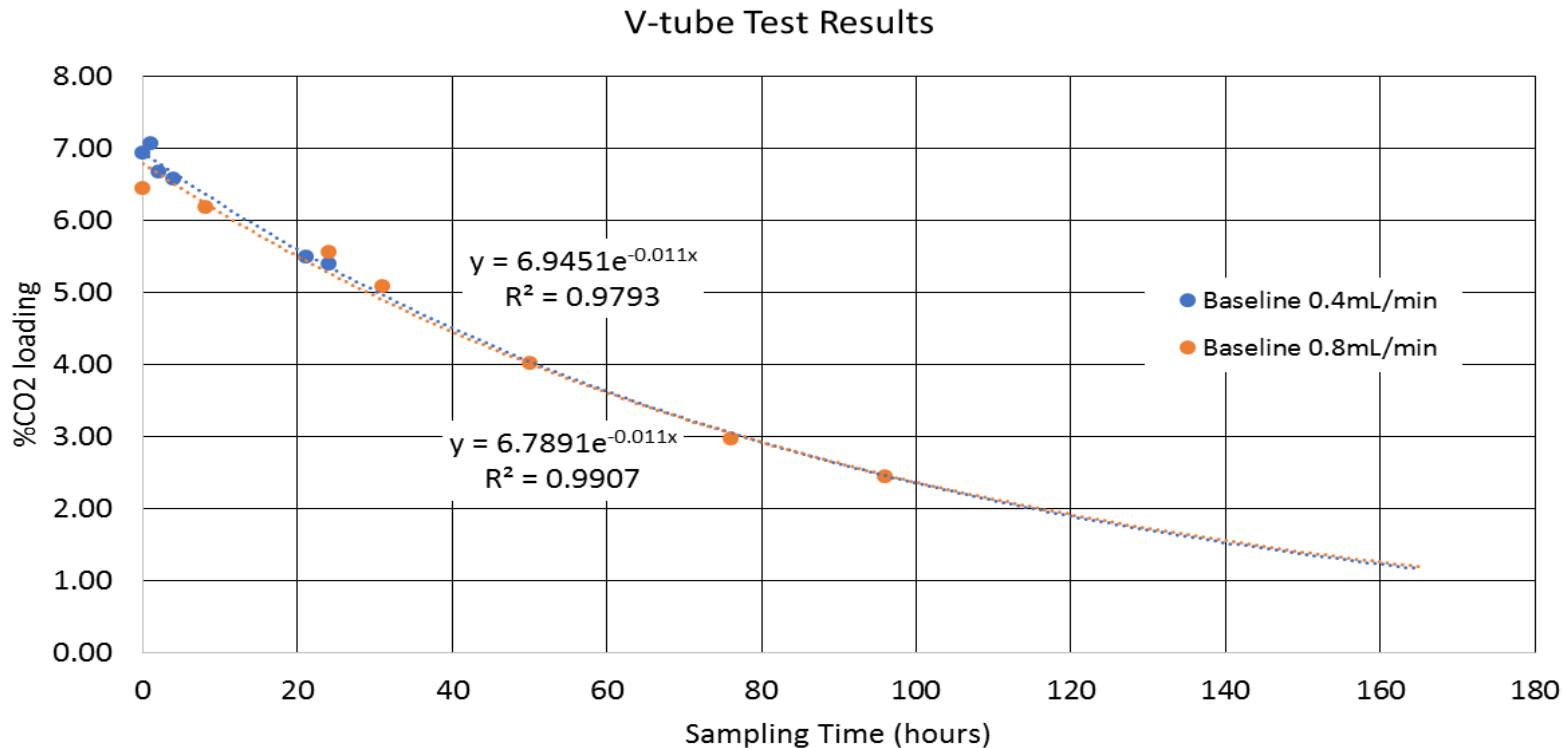
Results



Initial baseline – pure DGA, 85°C, ~0.4mL/min
flowrate, continuous flow sampling, no sweep gas



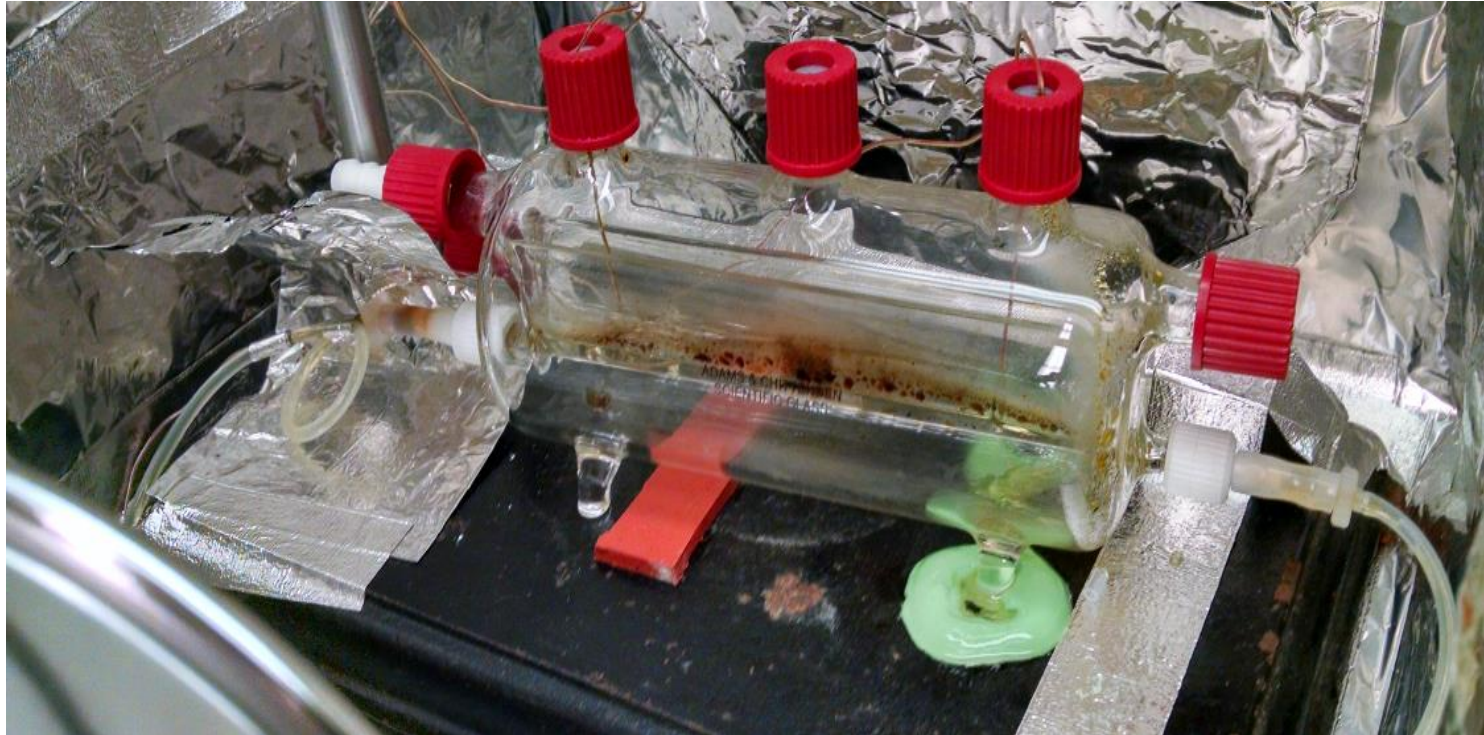
Results



Modified baseline – pure DGA, 85°C, ~0.8mL/min flowrate, continuous flow sampling, no sweep gas



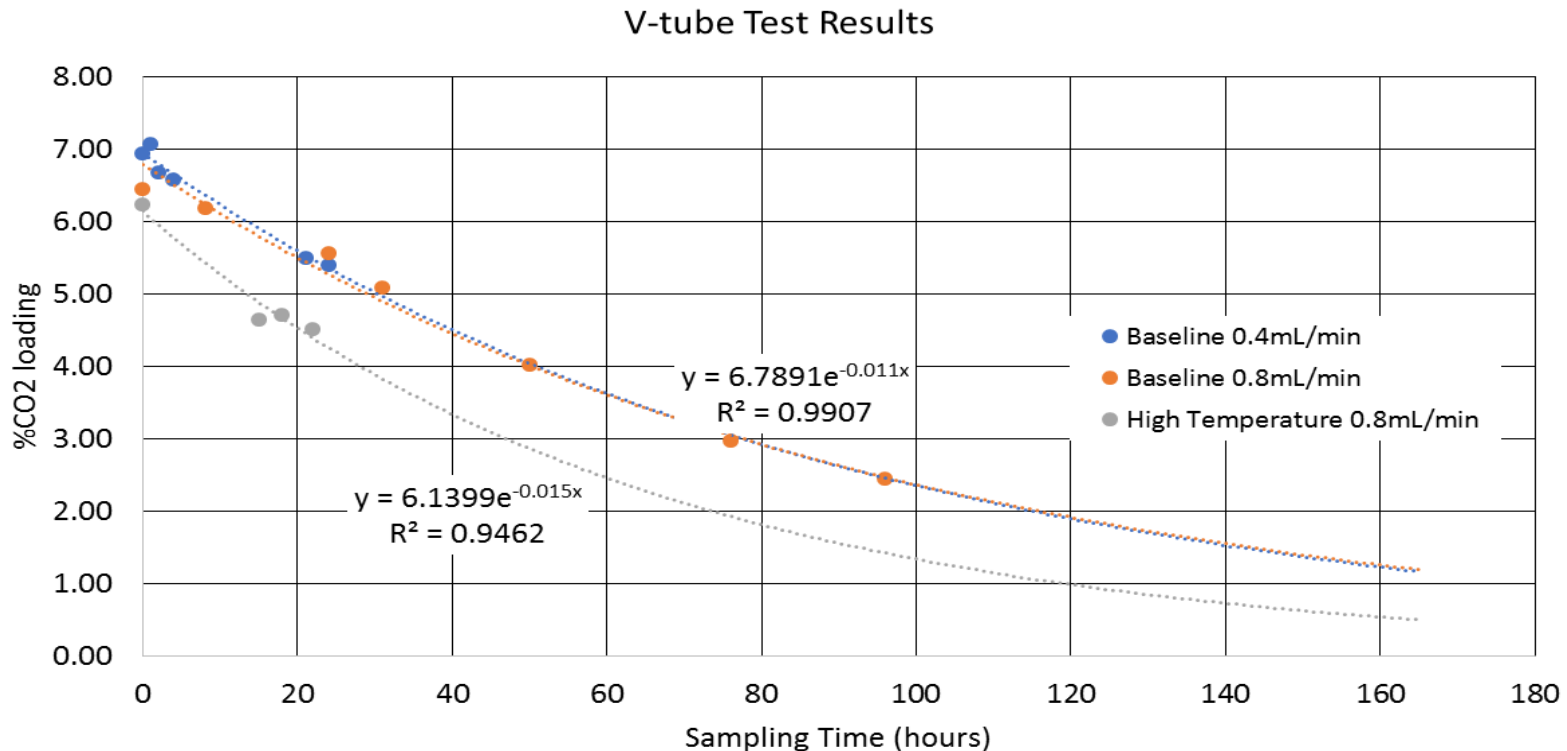
Results



Discoloration of evaporated (and re-condensed)
DGA



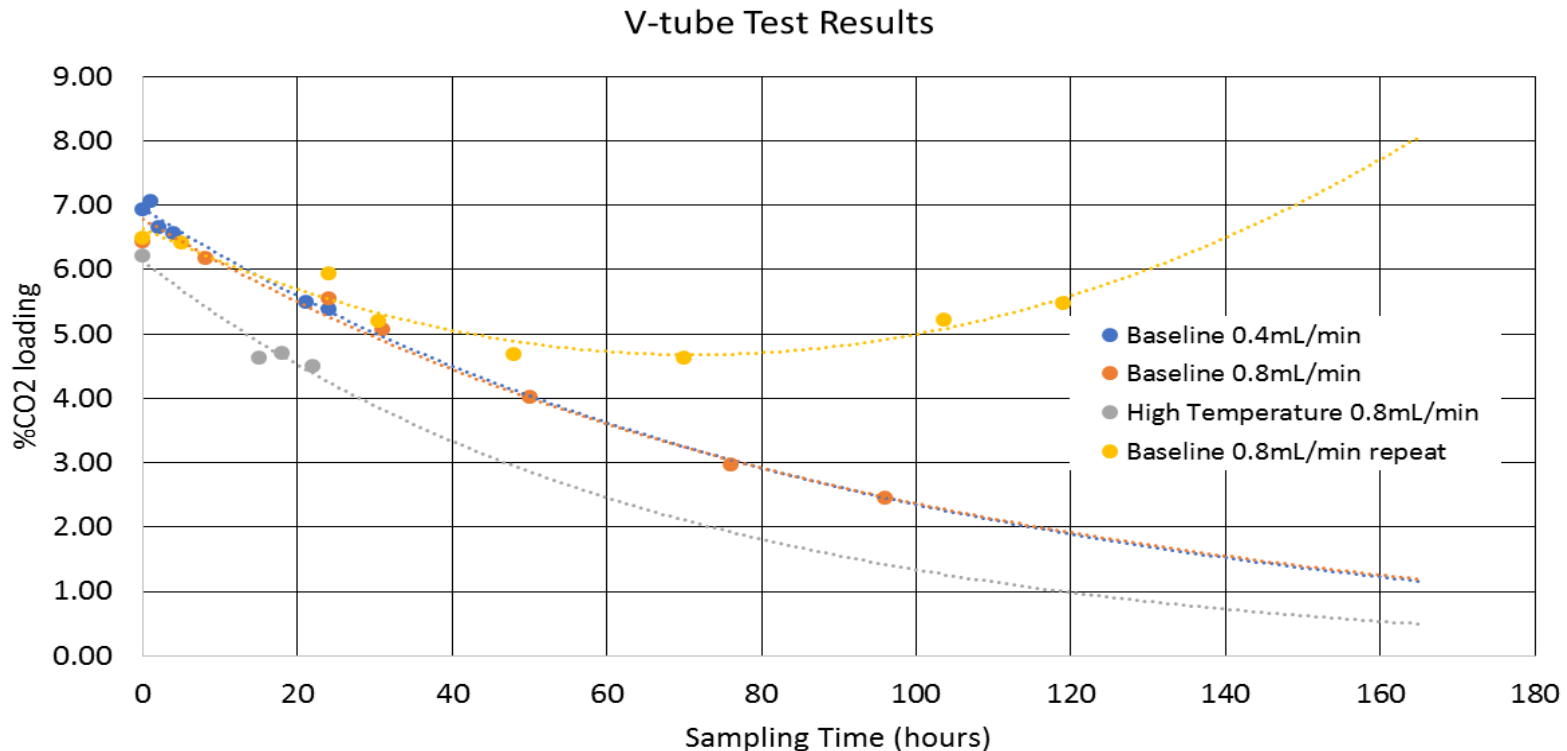
Results



Experimental – pure DGA, 105°C, ~0.8mL/min,
continuous flow sampling, no sweep gas



Results

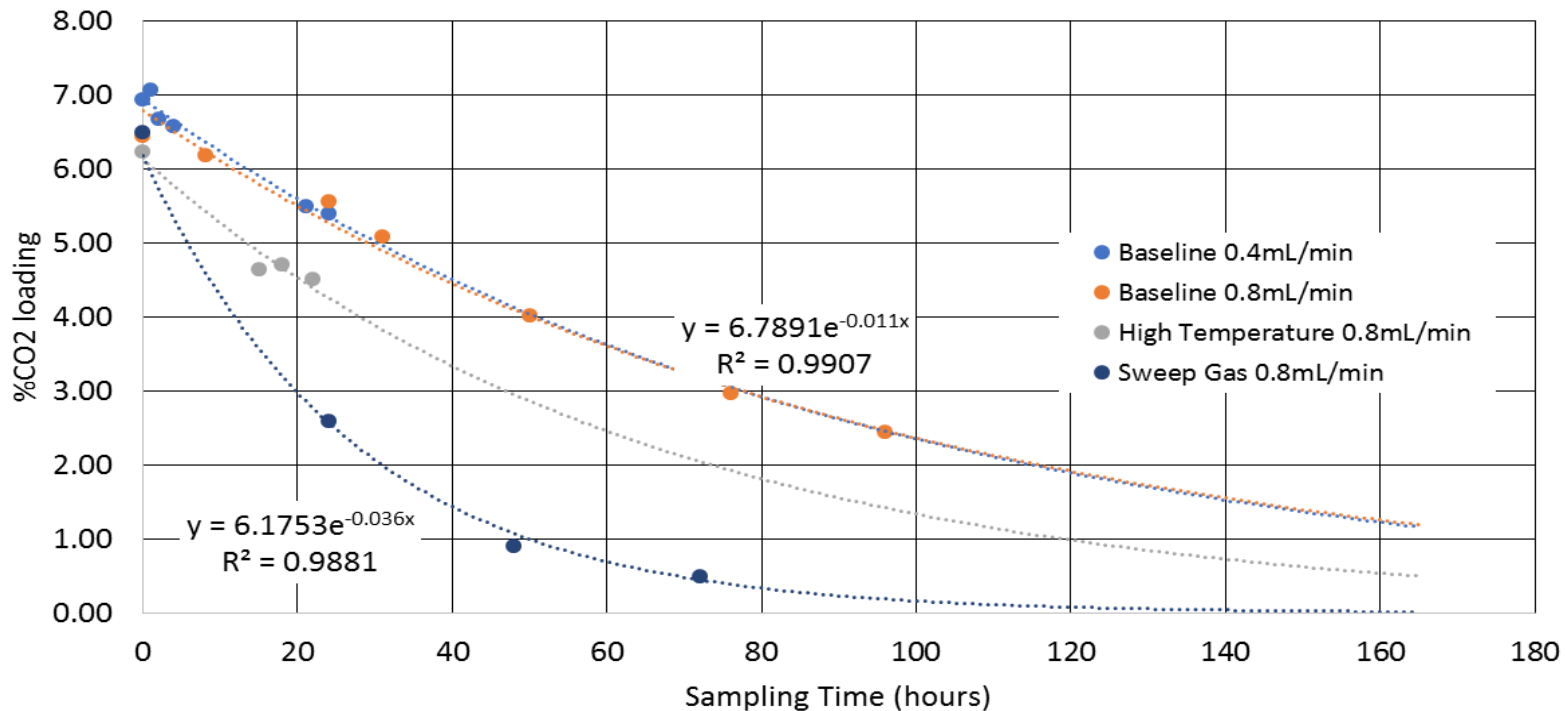


Baseline – pure DGA, 85°C, ~0.8mL/min,
continuous flow sampling, no sweep gas



Results

V-tube Test Results

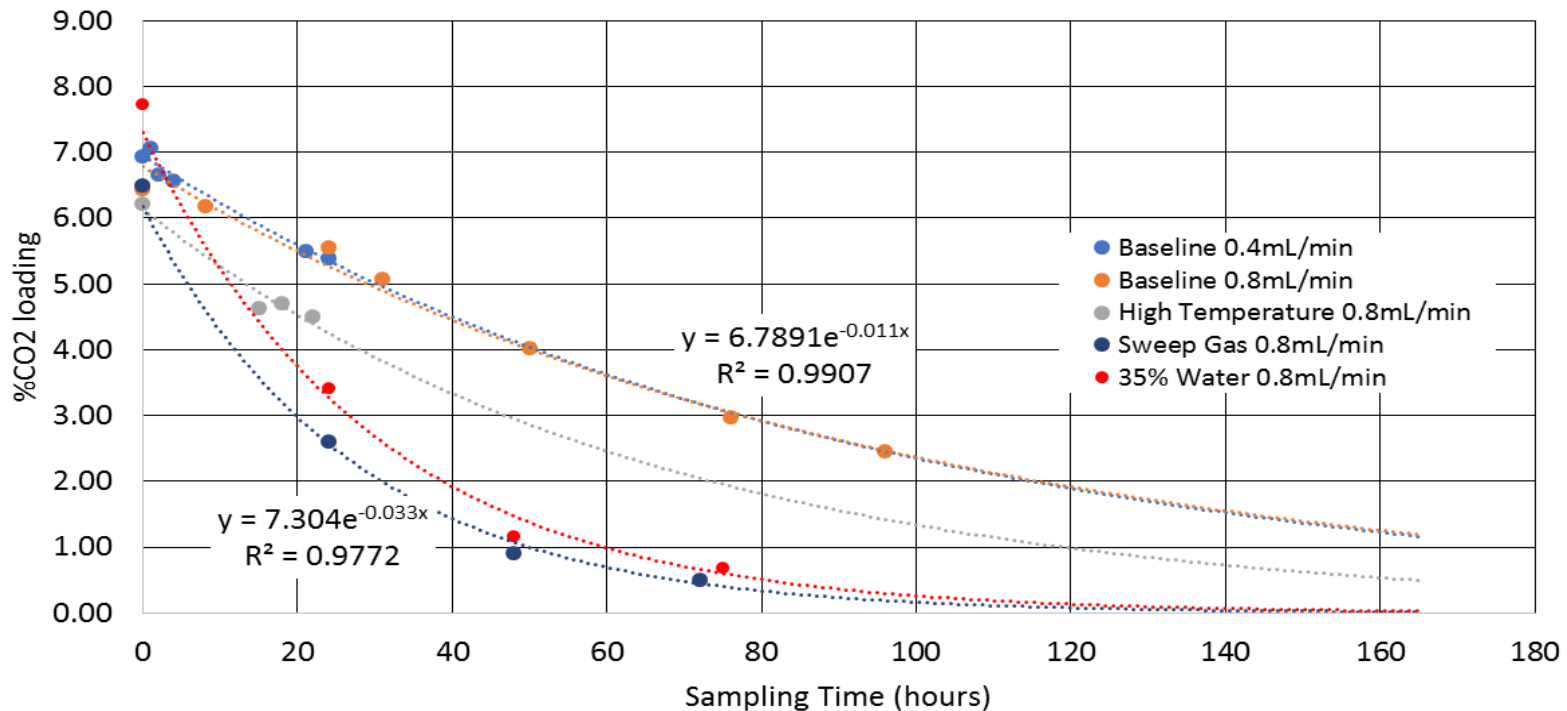


Experimental – pure DGA, 85°C, ~0.8mL/min,
continuous flow sampling, 400mL/min N₂ sweep
gas



Results

V-tube Test Results



Experimental – 35% H₂O + 65% DGA, 85°C,
~0.8mL/min, continuous flow sampling, no sweep
gas



Discussion and Current/Future Work

Moderate success using FTIR spectroscopy to analyze CO₂ % loading on DGA

- Repeatability
- Reducing sampling volume

35% H₂O + 65% DGA solution

- Single pass flow
- Scaling up
- Bubbling
- Re-capturing evaporated H₂O and DGA

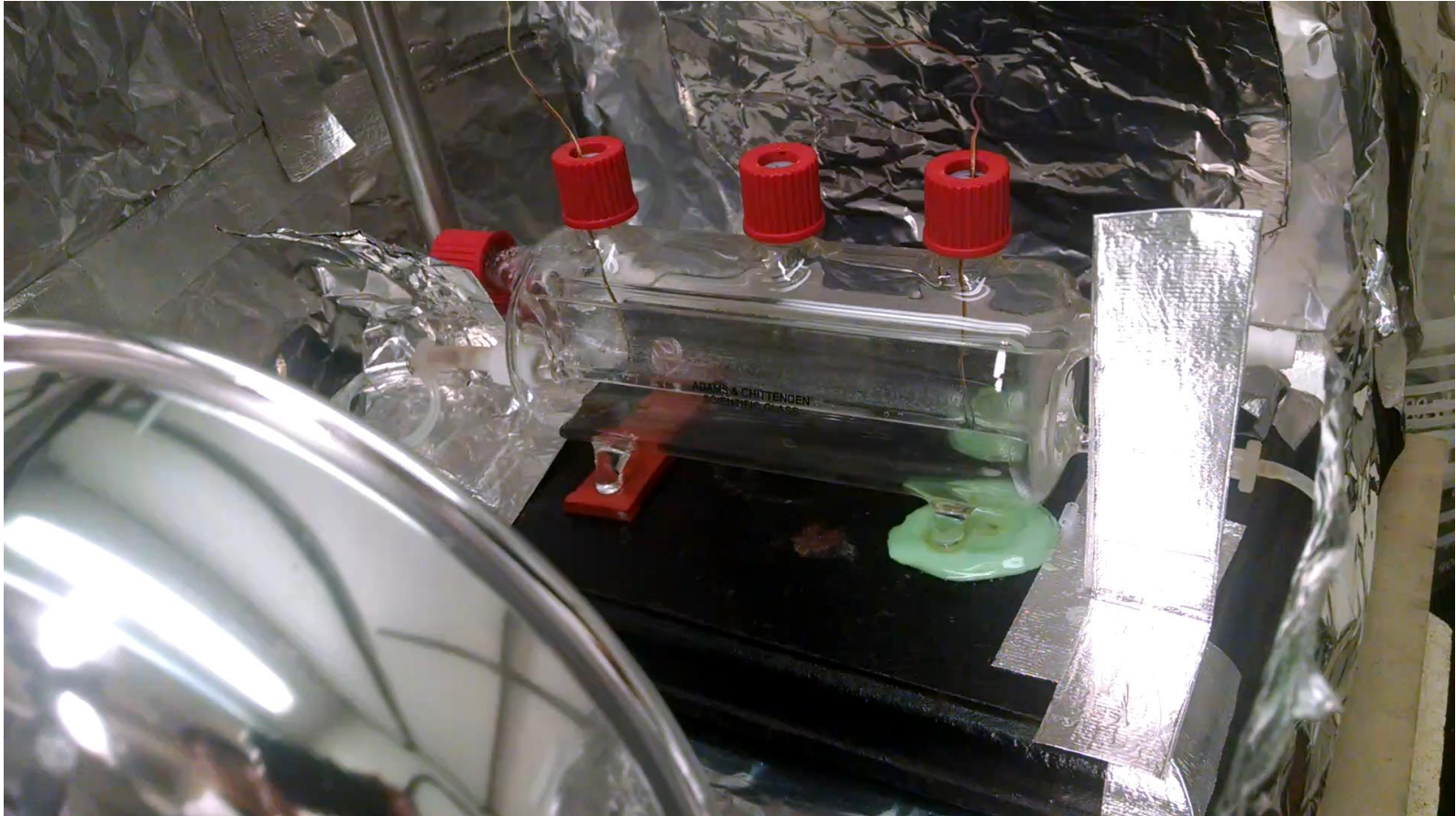


Acknowledgements

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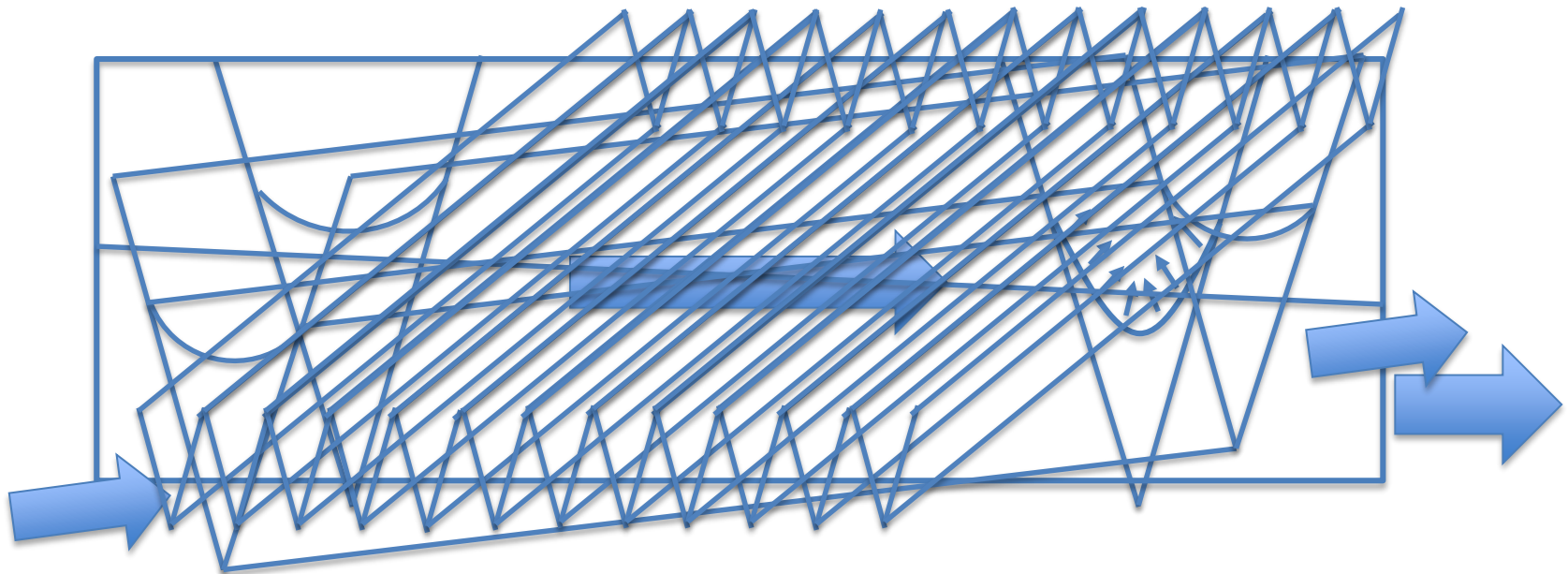


Backup Slides



Microgravity Adaptation

Potential design solution: “V-channel” direct air contactor



Degasser Design

Single-pass flow sampling

