Electronic Supplementary Material (ESI) for Lab on a Chip.

Supplementary Information for

A low-cost, non-invasive phase velocity and length meter and controller for multiphase lab-in-a-tube devices

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S-1: OVAL Validation

A custom-developed MATLAB image processing code in conjunction with a digital camera (LUMIX GH5, *Panasonic*) collecting video at 59.94 frames per second was used for automated phase detection, tracking, and measurement. Blobs corresponding to identified liquid slugs are tracked across the frame by the position of their bounding boxes, and the liquid slug length is determined from the line profile drawn through the center axis of the slug. Liquid slugs are dyed blue with a few drops of food dye for camera visualization. It should be noted that the presence or lack of dye does not change the ability of the phase sensors to detect liquid slugs. All analysis is conducted on the red channel of the image. A frame around 10 s into the video is selected as the background frame, and before applying the threshold to each other frame, this frame is subtracted from the one being analyzed to filter out stray shadows from the tube.

As the analysis progresses, the analyzed data transitions from being organized on a per-frame basis to being organized on a per-slug basis. First, the code identifies liquid slugs on each frame and identifies their positions. Then, with the help of their positions, the liquid slugs are identified across frames and a new MATLAB struct is populated with data pertaining to each slug. Image processing extracts the data about liquid slugs on each frame. Once the locations of liquid slugs are found, a liquid slug in a subsequent frame is identified as the same if its position is nearest and in the direction of motion (Figure S1).

Data for each **frame**
Frame number
Full cropped image
Binarized image
Image of complete slugs
For each **slug,** on frame
Centroid
Extrema
Bounding box
Number on frame

Data for each **slug**, all frames
Frames list
Addresses (position on frame) list
Times list
Pixel distance → distance
Velocity
Bounding box
Line profile→ length

Figure S1. *Data organization during image processing.*

The distance in pixels from each liquid slug's position to its first tracked position is calculated and converted to distance in mm with an image scale, which is set manually based on a ruler placed in the frame. The slope of the line of best fit for the distance *vs* time curve (*i.e.*, average velocity) is taken as the camera velocity – a process illustrated in Figure S2.

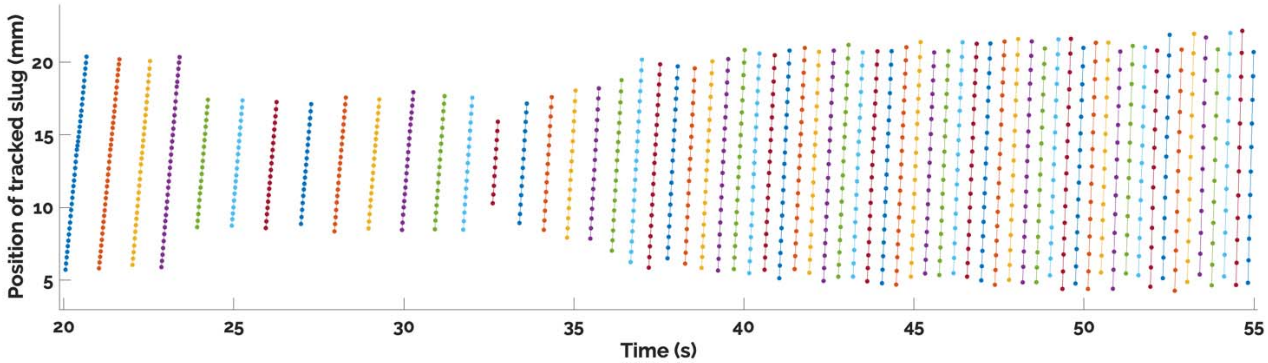

Figure S2*. Sample axial position vs. time of tracked slugs from validation run with a fluoropolymer tubing with an inner diameter of 0.02''. Each line corresponds to a different liquid slug. The shorter lines indicate longer liquid slugs, which necessarily move from edge to edge in fewer frames. Gas flow was set by a mass flow controller, and liquid flow was set with a continuous positive displacement pump.*

For each liquid slug in each processed frame, the outermost dimensions of the bounding boxes of the preceding and following liquid slugs as well as the current liquid slug's position are used to take the line profile brightness along the axis of the liquid slug. In a method mirroring that of the liquid slug detection algorithm, the average brightness inside and outside of the liquid slug is calculated, and the start and end of the liquid slug are determined by the position at which the line profile brightness crosses the average of the inner and outer brightness values (Figure S3). Note that a liquid slug must be fully within the field of view on both lagging and leading edges before identification.

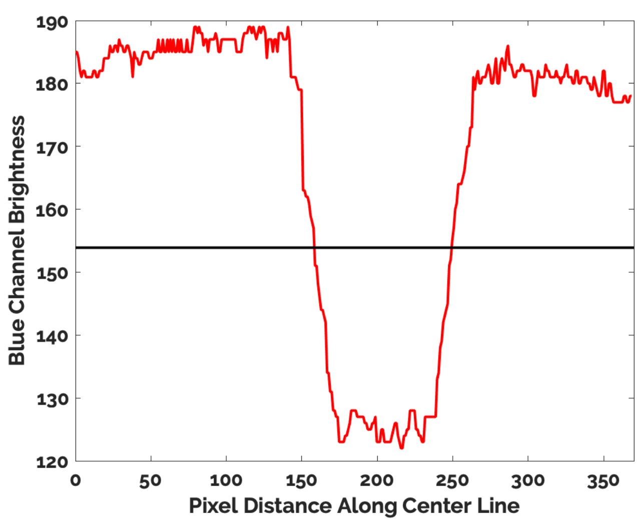

Figure S3*. Example line profile of a liquid slug (in red) and automatically determined threshold (in black) for liquid slug length determination of a liquid slug tracked within an FEP tubing with an inner diameter of 0.02''. Image scale was 0.0414 mm/pixel.*

S-2: Description of Real-Time Liquid Slug Detection

During flow characterization and control, samples were read from the optical sensors at a sample rate of 1000 Hz. From a running window of the last 2800 samples, a voltage threshold was set at half the maximum and minimum reading for each phase sensor. A typical sensor signal is shown in Figure S4 and has a range of about 2 or 3 V, depending on the sensitivity and internal device calibration. A dead zone spanning 0.4 V (*i.e.*, ± 0.2 V) around the threshold was used to prevent excessive triggering due to noise.

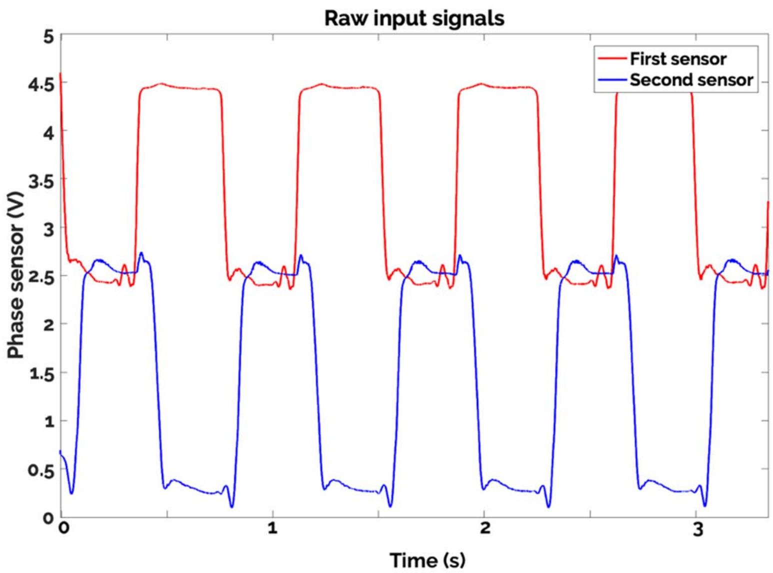

Figure S4. *Example voltage read-out of 2 phase sensors. Sample rate of example signal is 5,000 Hz. Flow rates were set with syringe pumps for water (10 mL 14.567 mm, SGE) and air (Steel 20 mL 19.13 mm). Liquid flow was 400 μL/min, and air flow was 600 μL/min.*

S-3: Additional OVAL Validation and Residual Plots

We validated the developed phase length and velocity meter/controller approach (*i.e.*, OVAL) for off-the-shelf fluoropolymer tubing with inner diameters (ID) of 0.02" and 1/16". A mass flow controller (Bronkhorst) and a continuous syringeless pump (M-6 series, Valco Instruments) were used to set the gas and liquid volumetric flow rates.

Figure S5 shows the residual plots for the pressure-driven validation in the main text. The mean absolute percentage error (MAPE) for the pressure-driven validation set is 1.3% for velocity and 3.3% for length.

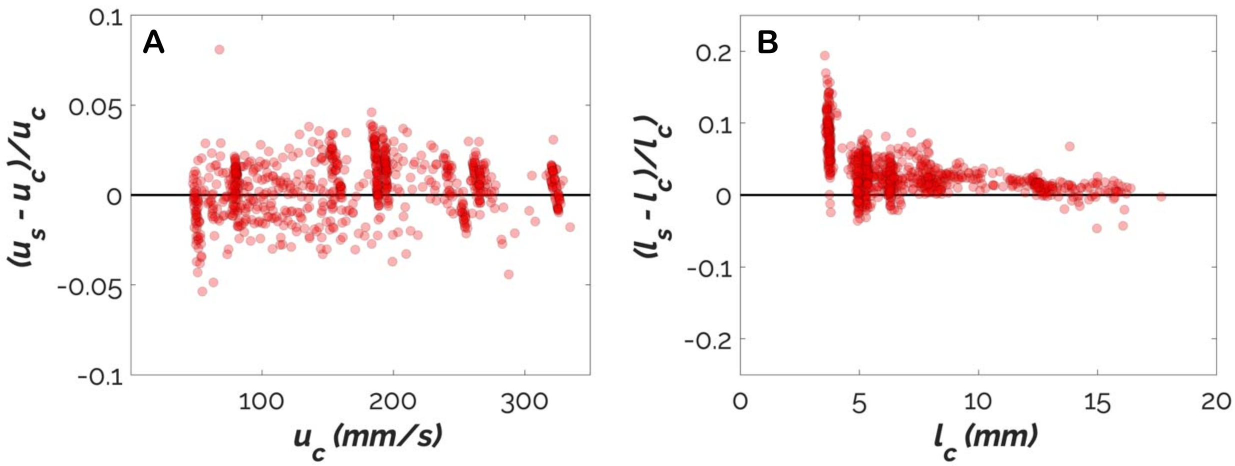

Figure S5. *Residual plot of (A) velocity and (B) length measurements as validated by digital camera slug tracking for the pressure driven flow system.*

Figure S6 shows the OVAL validation results for the fluoropolymer tubing with 0.02" ID. The validation flow rate ramps took place over approximately 160 s. The mean absolute percentage error is 1.4% for velocity and 2.0% for length.

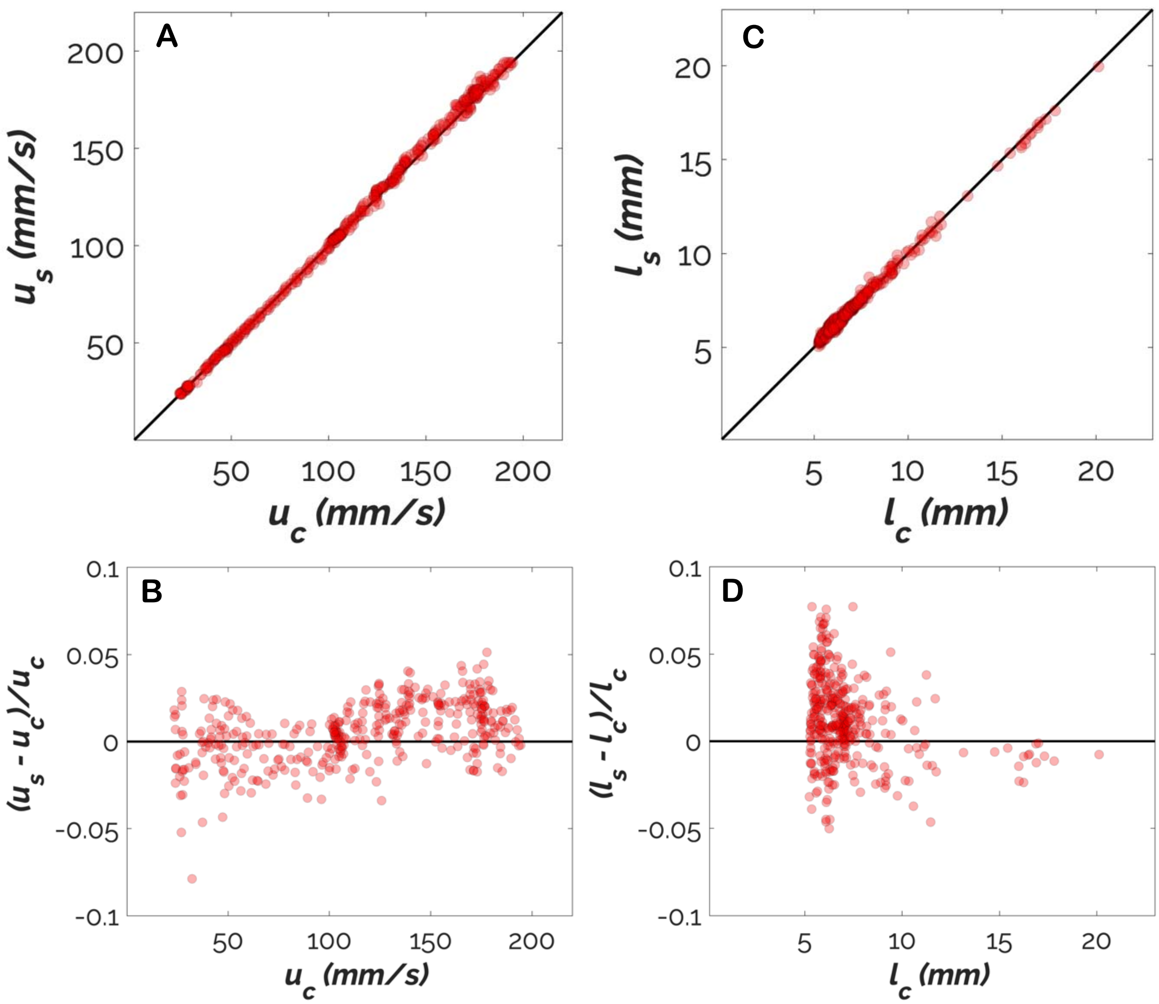

Figure S6. *Validation and residual plots for phase velocity (A, B) and phase length (C, D) for fluoropolymer tubing with 0.02" ID. Liquid (water and dye) flow rate spanned 84–346 uL/min. Gas (air) flow spanned 348–2000 uL/min. The video contained 9,600 frames.*

Figure the OVAL validation results for the fluoropolymer tubing with 0.0625” ID. The flow rate ramps took approximately 340 s. The mean absolute percentage error is 1.0% for velocity and 7.5% for length.

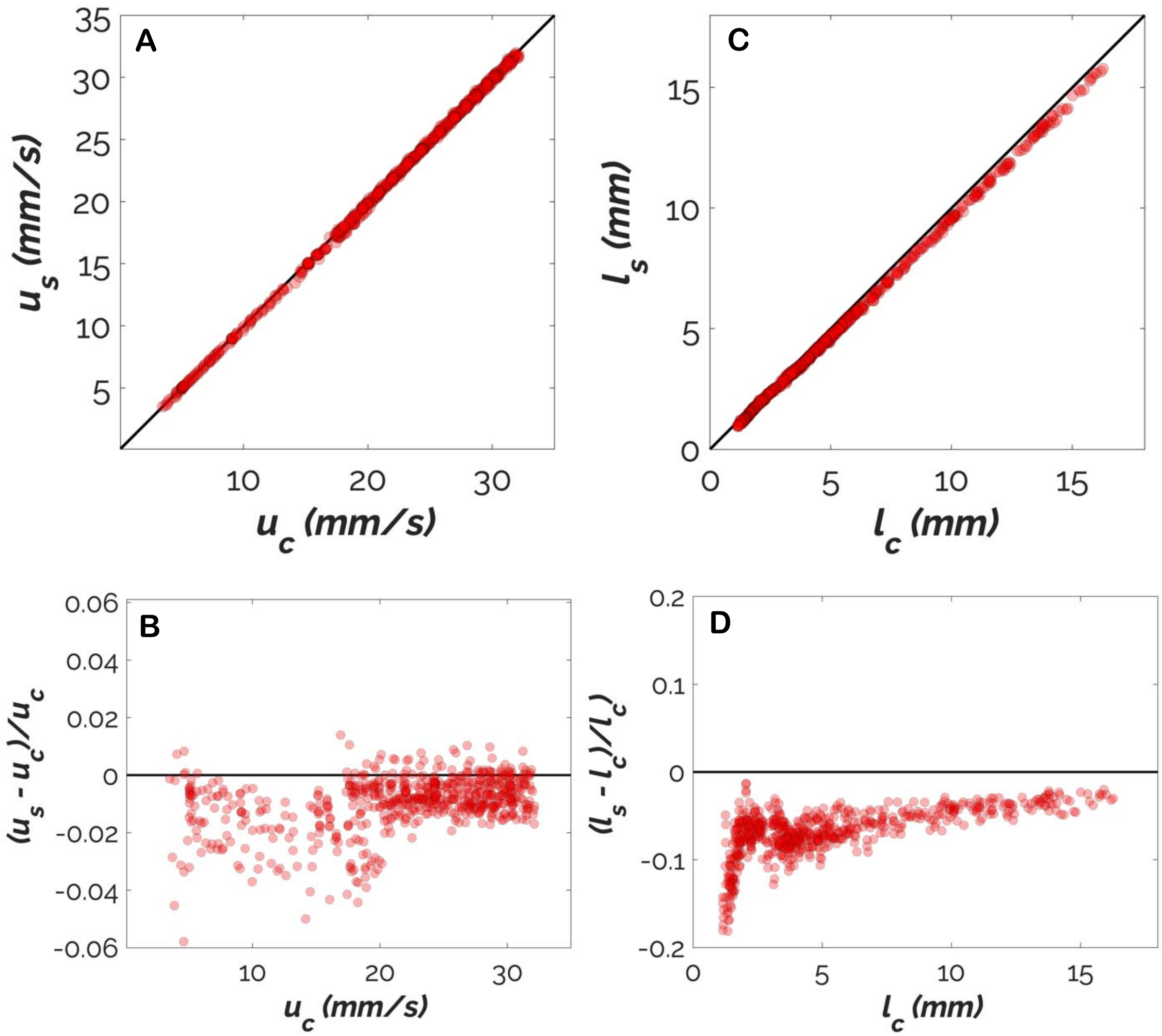

***Figure S7**. Validation and residual plots for phase velocity (A, B) and phase length (C, D) for fluoropolymer tubing with 0.0625” ID. Liquid (water and dye) flow rate spanned 75–2000 uL/min. Gas flow rate spanned 189–2000 uL/min. The video contained 21,450 frames.*

It should be noted that the sensitivity of the method is limited by the sample rate. Smaller, faster liquid slugs suffer from sampling error. A liquid slug would not be detected if $\frac{u}{l} < sr$, where sr is the sample rate in Hz.

S-4: Parameter Space Mapping

For the parameter space data collection, a custom-developed LABVIEW VI was used to repeatedly set random gas and liquid flow rates within a predetermined range, wait a fixed amount of time, and then re-initialized the flow by flushing the system with gas.

The history of velocity and length measurements for each condition was recorded, but only steady state values are incorporated into the parameter space. An example of the recorded data is shown in Figure S8. To determine flow stability, the average measurement and standard deviation of the last 12 s of data are used to calculate the "relative error," or average divided by standard deviation. Data points are accepted if both liquid slug velocity and slug length have relative error below their respective cutoffs. In this case, the cutoff was 0.01 for liquid slug velocity relative error and 0.05 for liquid slug length relative error. For the fluoropolymer tubing with 0.03'' ID, 650 runs were collected, and 423 points were included in the parameter space. This approach also highlights the ability of OVAL in evaluating stability of the multiphase flow in the microreactor, which may be adapted towards rapid reporting of flow segmentation regimes.

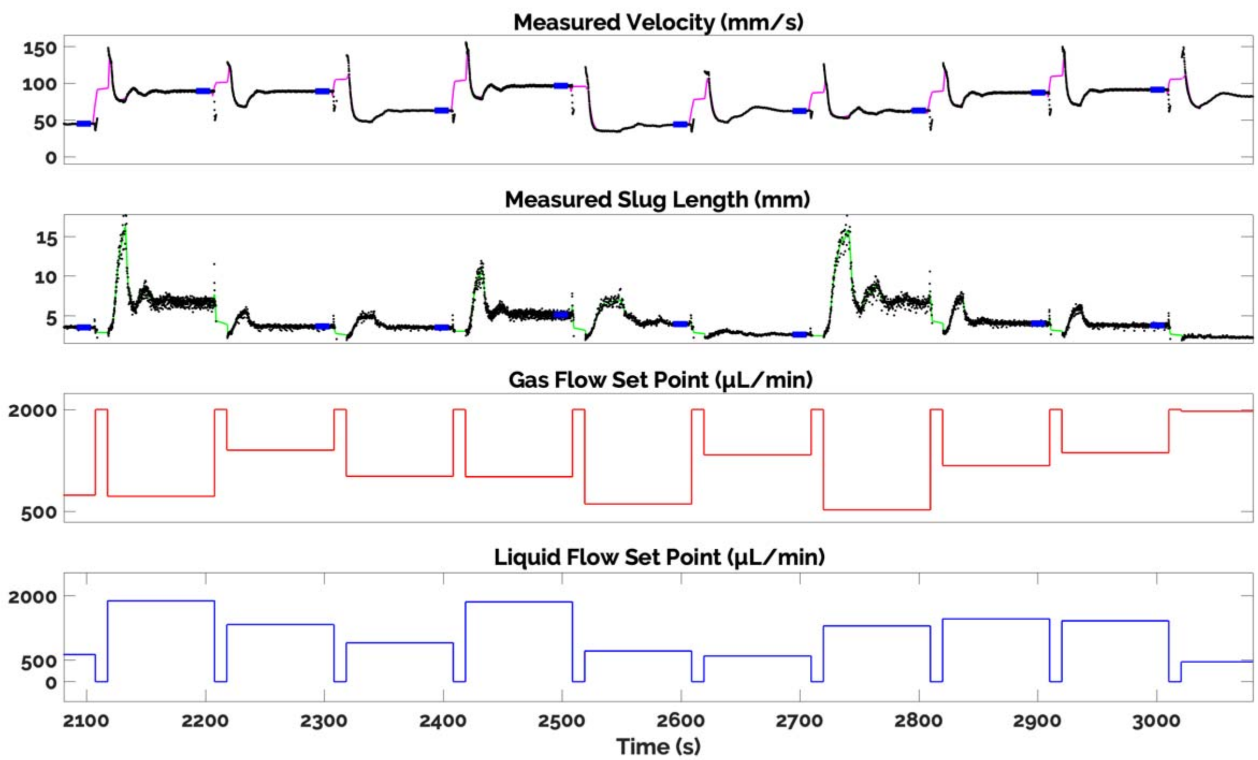

Figure S8. *Example of parameter space data extraction. Measurements are shown as black points; moving-average smoothed data are shown in pink and green for velocity and length. Dark blue bars on the liquid slug velocity and length plots represent regions of acceptable relative error spanning the relevant time and at the value of the average. Extracted data is matched with the appropriate gas and liquid flow setpoints to form the parameter space.*

The residuals shown in Figure S9 are calculated from the fitted models described in the main text. The fractional residual following the form (measured-predicted)/predicted is reported for the parameter space. Discrepancies from the model may occur because these models may not account for factors pertaining to the specific segmentation regimes present in the employed T-junction.

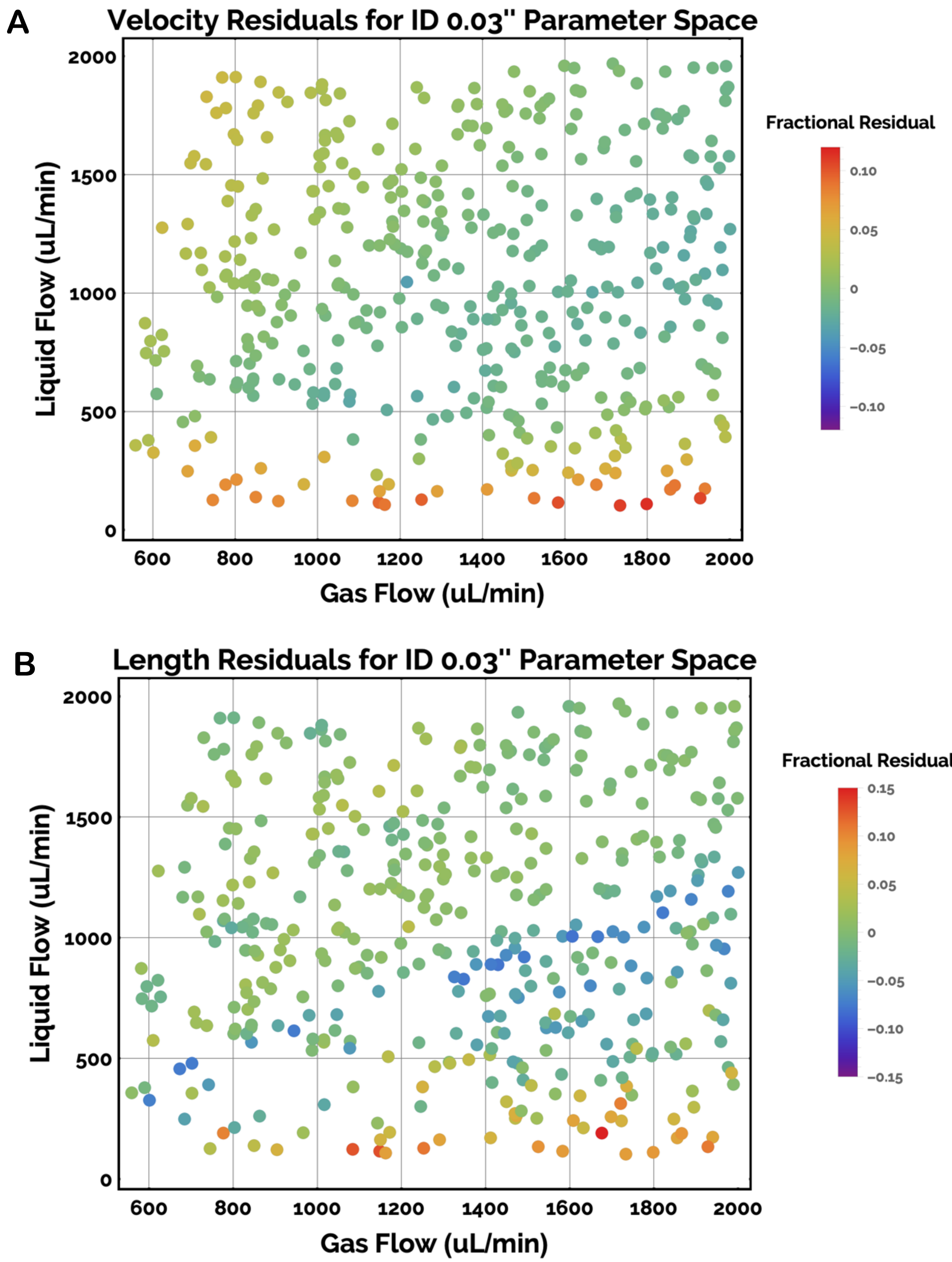

Figure S9. *Fractional residuals of the liquid slug (A) velocity and (B) length compared to a model of the system, where fractional residual is defined as (measured – model)/model. The tube inner diameter was 0.03''.*

S-5: Process Controller Descriptions

The PID control was implemented using a built-in LabVIEW VI ("PID Autotuning"). Autotuning variation was used to determine the optimal control parameters for the system of $K_c = 92$, $\tau_I = 0.5$, and $\tau_d = 0.01$.

The two-input, two-output fuzzy logic system (TITO FLS) was developed in the LabVIEW Fuzzy System Designer, and it uses a combination of linear and Gaussian functions, shown in Figure S10 where NN, N, Z, P, and PP correspond to "very negative", "negative", "zero", "positive", and "very positive" membership groups, respectively. Centroid fuzzification and defuzzification was used in direct relationships between the velocity error and net flow rate change and the slug length error and input liquid fraction change – i.e. *IF Velocity Error is X THEN Net Flow Rate Change is X AND IF Slug Length Error is Y THEN Input Liquid Fraction Change is Y* where *X* and *Y* correspond to arbitrary membership functions. Repeated iterations of the feedback control loop incorporated a delay time, which was calculated from the sampling distance (k) of 5 cm and the last measured slug velocity (u_{last}) according to the relationship $t_{delay} = k/u_{last}$.

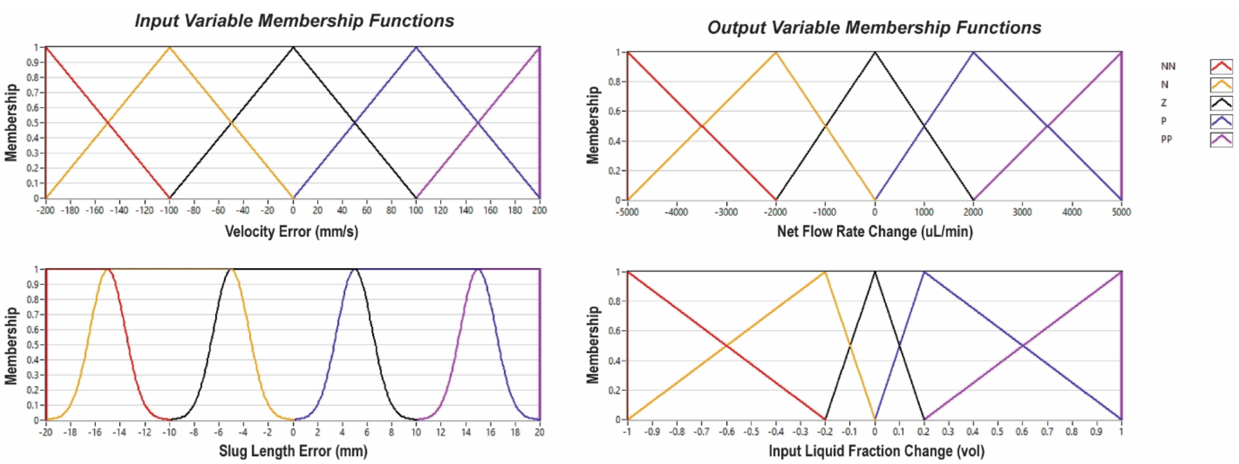

Figure S10. *TITO Fuzzy Logic controller membership functions.*