✨AI for Samples✨:

🤖Automated Injection for fast and efficient sample loading

Charmaine Lui [1,2] – charmaine.lui@sjsu.edu, Bob Sublett [1], Sebastian Dehe [1], Raymond G. Sierra [1]

1. SLAC National Accelerator Laboratory, USA   2. San Jose State University, USA

SLAC NATIONAL ACCELERATOR LABORATORY   U.S. DEPARTMENT OF **ENERGY** | Stanford University   **SPARTAN SPARK**

Upgrading Sample Loading Efficiency

The LCLS-II HE upgrade will enable faster experiment runs, which means more samples can be tested within a given timeframe. Stopping frequently to manually switch in new samples would become a significant limiting factor for beamtime efficiency [1].

Autosamplers are used in HPLC systems for automated sequential injection from tray of sample vials (e.g. 70 vials of 1.5 mL). Replacement of single reservoirs with an autosampler could support high-throughput of many different samples with the same solvent.
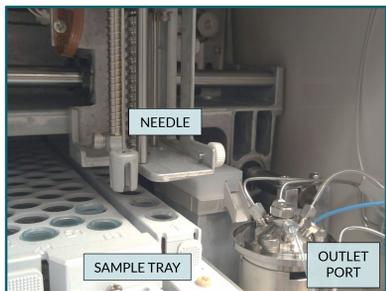

Fig. 1 Autosampler needle & tray with sample vials.
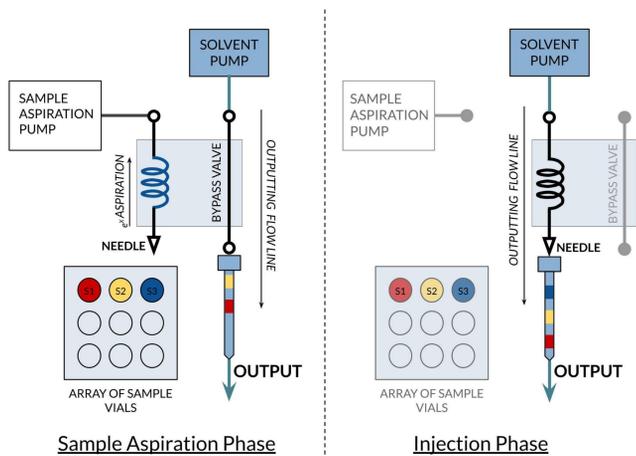
Autosampler Supports Wide Parameter Ranges

Fig. 2 Diagram representing a basic autosampler operation cycle: sample aspiration (intaking of sample into the needle storage loop), followed by injection (pushing of sample to output and rinsing of needle storage loop with solvent).

- Aspiration volume of <50 µL or <2 mL per aspiration-injection cycle (storage loop dependent)
- 3 rinse options (pre-aspiration, post-injection, both)
- Sample tray options (1, 1.5, 4 mL vials; MTP; DWP) [2]
- Optional temperature control system

Autosampler Supports Speeds of ~1 Sample/min

- Benchmarking: manual reservoir swap can take up to ~30 min [1]
  - *Automated swap = $t_{movement}$ + $t_{aspiration}$ + t_{wash}*
  - Movement time ($t_{movement}$) per cycle:
    - <10 s without external needle wash 'pit stop'
    - <18 s with 'pit stop'
  - Aspiration time ($t_{aspiration}$) = ~3.5 s for filling a 50 µL storage loop at 15 µL/s (0.9 mL/min)
  - External needle wash (t_{wash}) between 0 to 60 s
- $t_{injection/rinse}$ dependent on solvent pump flow rate
  - Can be run for flow rates between <5 µL/min and 10 mL/min (pump dependent)

Sample Dispersion Affects Local Concentration

- The flow through the lines smears out initially sharp concentration profiles (Dispersion), which can lead to dilution and cross-contamination.
- At low flow rates, immiscible fluids (e.g. air or oil) are effective for separating sections of liquid.
- Problems:
  - Air division to prevent tail-end dispersion traps residual sample in the loop, creating a trailing line of cross-contaminated samples
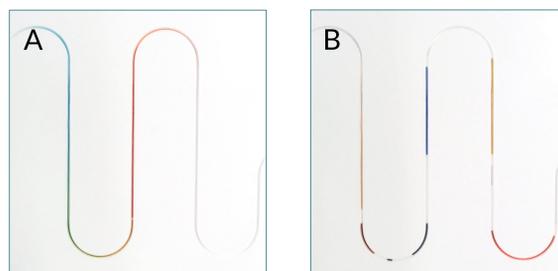 - Air insertion by design is not effective at high flow rates (e.g. 0.6 mL/min)

Fig. 3 Sequential 10 µL samples of red, yellow, and blue dye in water without air gaps (A), and with air insertion of 10 µL (B). Irregular air gaps appeared at the outlet, possibly introduced at the switch valve.

Distinct Sequential Samples, Rayleigh Jet Use Case

- Delivery to 30 µm ID glass nozzle, Q = 0.6 mL/min
- 3 sequential 10 µL samples of water with red, yellow, & blue dye; water as the flow line solvent; no air gap
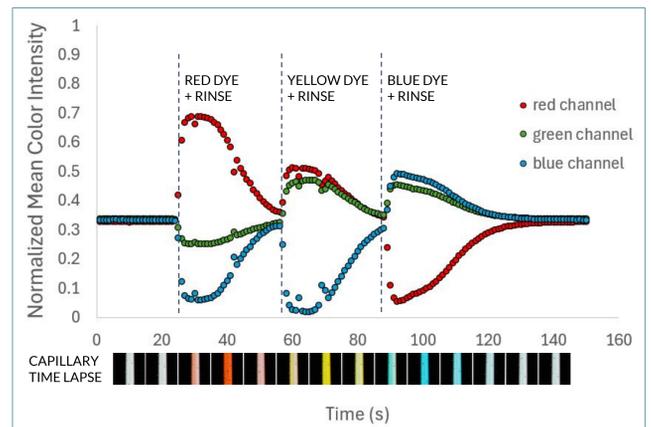

Fig. 4 Normalized mean color intensity of Rayleigh jet for the described sample sequence.

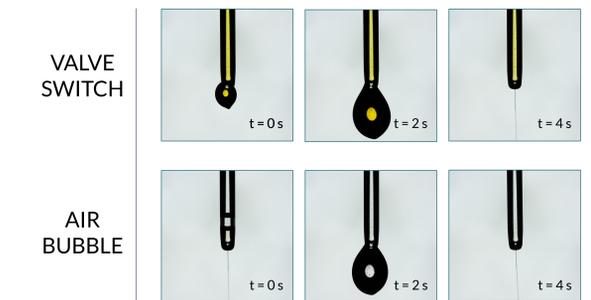## Jet Recovers from Autosampler Perturbations

Fig. 5 Output from a Rayleigh jet, showing jet recovery from temporary flow disruptions due to valve switching (middle) or 1 µL air bubble insertions (bottom).

The Future of Automated Injection

- Autosamplers can provide efficient sample exchange to support high rep. rate experiments (140 kHz) (~ 2·10^6 shots per 10 µL sample; 14 m/s, Δ 100 µm).
- Next steps:
  - Stable air insertion or other divisions against dispersion; UV-VIS for concentration changes
  - Compatibility with differing delivery methods, e.g. GDVN jets, vacuum chambers
  - Testing with crystals; viscosity & clogging; settling

REFERENCES

1. Sierra, R., Dehe, S., Armenta, R., Goodrich, H., & Olsen, K. (2025, June 26). *High repetition rate Sample Environment and Delivery for biology in LCLS-II-HE* [PowerPoint slides].
2. Shimadzu Corporation. (2020). SIL-20A/20AC SIL-20AHT/20ACHT Instruction Manual.