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**Article title**

*Please avoid acronyms and abbreviations where possible.*

**Closed-loop Spectroscopy Lab – Liquid: A Chemistry “Hello, World!” for Self-driving Laboratories**

**Authors**

*List all authors. Please mark the corresponding author with (\*)*

Sterling G. Baird1\*, Taylor D. Sparks1

**Affiliations**

1Materials Science & Engineering, 122 S. Central Campus Drive, #304 Salt Lake City, Utah 84112-0056

**Corresponding author’s email address and Twitter handle**

*Institutional email address preferred. If you have a Twitter handle, please add it here ‘twitter: @....’*

[sterling.baird@utah.edu](mailto:sterling.baird@utah.edu)

@SterlingBaird1

**Abstract**

*Max. 200 words. Remember that the abstract is what readers see first in electronic abstracting & indexing services - make it brief, specific, interesting and easy to understand. If a research article refers to your hardware, cite that research article here.*

Materials acceleration platforms have the potential to reduce cost- and time-to-market of new materials from $20 million and 20 years to as little as $1 million and 1 year; however, the capital and expertise is often large. Previously, we introduced the idea of a minimal working example for a self-driving laboratory that used red green blue light-emitting diodes and a discrete-channel spectrophotometer in conjunction with optimization algorithms to match target spectra. Here, we extend this idea to a materials optimization problem using food dye and performing color matching using the same spectrophotometer. This optimization task costs less than 300 USD, requires less than three square feet of desk space, and less than three hours of total setup time from the shopping cart to the first “autonomous drive.” The demo is modular and extensible, designed such that inexpensive, chemically susceptible parts can be replaced with more expensive, chemically resistant equivalents and the food dye replaced with application-specific chemicals. This demo serves as a steppingstone towards larger, high-impact studies with low startup costs. For example, formulations of up to 16 distinct liquid battery or fuel cell electrolytes can be optimized for ionic conductivity properties given additional pumps, an appropriate sensor, and peripherals.

**Keywords**

*Add at least 3 keywords and a maximum of 6 keywords.*

Self-driving laboratory, chemistry automation, Bayesian optimization, cheminformatics, materials informatics, internet of laboratory things

**Specifications table**

*Please replace the italicized instructions in the right column of the table with the relevant information about your hardware.*

|  |  |
| --- | --- |
| Hardware name | *The name of the hardware that you have invented/customized* |
| Subject area | * Chemistry and biochemistry |
| Hardware type | * Measuring physical properties and in-lab sensors |
| Closest commercial analog | Cary 3500 UV-Vis Spectrophotometer with Cary Sipper Flow Cell Pump accessory (though our demo has much lower resolution) |
| Open source license | *All designs must be submitted under an open source license (for more details see the* [*Guide for Authors*](https://www.elsevier.com/journals/hardwarex/2468-0672/guide-for-authors)*). Please specify the open source license you’ve selected here.*  CERN-OHL-P-2.0 |
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1. **Hardware in context**

Materials acceleration platforms have the potential to dramatically accelerate the understanding and discovery of new materials 1–13; however, the barrier-to-entry can be prohibitive to researchers. Low-cost platforms for teaching and prototyping help reduce this barrier 14–19. Previously, we proposed an idea for a low-cost, low footprint, and low setup time self-driving laboratory that performed light-based color matching via a discrete-channel light sensor and red green blue (RGB) light-emitting diodes20. The demo captures many key principles of a self-driving laboratory (sending commands, receiving experimental results, and performing physics-based simulations and active learning); however, no chemistry or materials science concepts were directly involved. Here, we propose an idea for an extension kit to the light-based color matching demo: an optimization task for less than 300 USD, three square feet of desk space, and three hours of total setup time. This allows for modular reuse of both hardware and software from the light-mixing demo, including more advanced topics such cloud experimentation and database logging. This demo, which we will refer to as Closed-loop Spectroscopy Lab: Liquid or CLSLab-Liquid, uses peristaltic pumps, a spectrophotometer, a cuvette, and a white light-emitting diode light source to perform spectrophotometry on a mixture of dilute red, yellow, and blue food coloring. We believe our demo meets the definition of a materials acceleration platform10:

[A system that] carries out high throughput and/or automated experiments, the results of which are fed back into the AI that guides the selection of subsequent rounds of experimentation to optimize or make a discovery.

Similar systems have been developed which involve the mixing of multiple liquid channels and measuring sensor property data. Roch et al. created a Bayesian Optimization Bartender (“Bob”) and similarly performed color-matching experiments using pumps and an RGB color sensor, though in a semi-autonomous fashion.21 Gutierrez et al. modified a 3D printer to create an automated liquid handler and monitor the evolution of oil droplets18. In follow-up work, Caramelli et al. performed color-matching experiments with peristaltic pumps and by monitoring solution colors with a webcam14. Keesey et al. developed a low-cost automated liquid-dispensing robot for $710 that has four dispensing channels.22 Other examples include semi-automatic and automatic titration experiments23–26, food formulation optimization27, 3D print parameter optimization28, syringe pumps29,30 and other liquid handlers31–33. In the specific case of automated mixing of liquid precursors and performing spectrophotometry, Agilent Technologies has a product and accessory called the [Cary 3500 UV-Vis Spectrophotometer](https://www.agilent.com/en/product/molecular-spectroscopy/uv-vis-uv-vis-nir-spectroscopy/uv-vis-uv-vis-nir-systems/cary-3500-uv-vis-spectrophotometer) and [Cary Sipper Flow Cell Pump](https://www.agilent.com/en/product/molecular-spectroscopy/uv-vis-uv-vis-nir-spectroscopy/uv-vis-uv-vis-nir-accessories/cary-sipper-flow-cell-pump), respectively. Our demo is effectively a low-cost teaching demo version of this higher cost platform, albeit at a much lower resolution.

1. **Hardware description**

*Describe your hardware, highlighting the customization rather than the steps involved in the procedure. Explain how it differs from other hardware and the advantages it offers over pre-existing methods. For example, how does this hardware compare to other hardware in terms of cost or ease of use, or how can it be used to develop further designs in a particular area?*

CLSLab-Liquid uses the pumping of dilute colored food dye via peristaltic pumps into a transparent cuvette chamber to perform low-resolution spectrophotometry. The hardware is accompanied by software to carry out fully autonomous color-matching experiments, including “Hello World” style tutorials for optimization and hardware-software interfaces. Additionally, the modular MicroPython code allows for easy reconfigurability to other problems, and the hardware can be replaced with chemically resistant materials for real-world optimization tasks. The demo was designed in a way that maximizes the optimal trade-offs between expense and expertise required. In terms of electronics expertise, only minimal soldering is required, and no custom parts (e.g., 3D-printed parts) are required. The system is closed, meaning it is resistant to external gases entering the system, which can be important for e.g., battery applications. Our setup is ideal for teaching and prototyping settings. A diagram of the system is given in Figure 1.

A picture containing application

Description automatically generated

Figure 1. Visual summary of Closed-loop Spectroscopy Lab – Liquid (CLSLab-Liquid). IV-style bags containing red, yellow, and blue food dyes and rinse water are connected to peristaltic pumps that feed into an optical cuvette and out into a waste bag. A white LED shines through the cuvette, illuminating a spectrophotometer that measures eight discrete wavelengths. The peristaltic pumps are operated via motor drivers which are controlled by a Pico Wireless microcontroller.

While CLSLab-Liquid shares similarities in terms of color-matching to that of the “Bayesian optimization Bartender” by Siefrid et al.21, it is also distinct in that it can be operated with many iterations in a fully autonomous fashion. Relative to Caramelli et al.14, CLSLab-Liquid uses up-to-date, user-friendly, and easily available (as of Feb 2023) hardware and software via the Pico Wireless and MicroPython ecosystems, respectively. The use of an optical cuvette and a many-channel spectrophotometer likewise creates a more direct companion to existing real-world UV-Vis spectrophotometry equipment. While there is an OEM product by Agilent Technologies, to the knowledge of the authors, this system is not at a price point amenable to classroom settings nor is it open source. Our device captures the key components of Agilent’s [Cary 3500 UV-Vis Spectrophotometer](https://www.agilent.com/en/product/molecular-spectroscopy/uv-vis-uv-vis-nir-spectroscopy/uv-vis-uv-vis-nir-systems/cary-3500-uv-vis-spectrophotometer) and [Cary Sipper Flow Cell Pump](https://www.agilent.com/en/product/molecular-spectroscopy/uv-vis-uv-vis-nir-spectroscopy/uv-vis-uv-vis-nir-accessories/cary-sipper-flow-cell-pump) combo in a low-resolution, low-cost setting. Additionally, CLSLab-Liquid is relevant to many chemistry applications where batch- or flow-reactor experiments are required since it can be operated in an iterative fashion with rinses in-between or in a continuous flow setting, respectively. Additionally, the use of peristaltic pumps rather than syringe pumps29,30 allows for large storage capacity and therefore a greater number of iterations (or longer continuous flow runtimes) before manual replacement of stock materials is required. However, more calibration may be required relative to a syringe pump. While liquid handlers are useful and could be used to perform the same color-matching demo, they often require either greater monetary expense, longer setup times, or more advanced user expertise. Additionally, liquid handlers generally do not mimic flow-reactor settings characteristic of industry and typically have harder constraints on the number of iterations available in batch-reactor settings due to a limited number of positions or containers available.

The hardware can be used to develop further designs in this area in applications such as:

* Titration experiments (optimizing pH)
* Optimization of ionic conductivity for battery electrolytes
* Solid-based color-matching

*Add 3-5 bullet points which broadly explain to other researchers - inside or outside of the original user community - how the hardware could help them, with either standard or novel laboratory tasks.*

Broadly, the hardware can help users in the following ways:

* Use to teach autonomous laboratory principles in chemistry and materials science courses
* Use as a proof-of-concept or prototyping platform for grant proposals
* Use as an optimization benchmark for comparing algorithm performance
* Use as part of a chemistry or materials data science hackathon

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*Your design files should be editable - see* [*OSHWA’s open source definition of ‘Documentation’*](https://www.oshwa.org/definition/) *for further details. You must then either:*

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* *Upload your design files as supplementary materials (e.g., CAD files, videos…) to Hardware X’s online editorial system when you submit your manuscript.*
* *Include your design files in the body of the manuscript (e.g., as figures).*

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*3D printing. Supplementary files that facilitate digital replication of the devices are encouraged; for example, STL files for 3D printing components. We recommend uploading CAD files to the* [*NIH 3D Print Exchange*](http://3dprint.nih.gov/) *as Custom Labware and then entering the link here.*

*Electronics: PCB layouts and other electronics design files can be uploaded to the* [*Open Hardware Repository*](http://www.ohwr.org/)*or other repositories or as supplementary materials.*

*Software and firmware***:** *All software files used in the design and operation of the hardware should be included in the repository. Provide a description of the software and firmware and use extensive comments in the code.*

1. **Design files summary**

*Complete a separate row for each design file associated with your hardware (including the primary design files). Any empty rows should be deleted.*

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| --- | --- | --- | --- |
| **Design file name** | **File type** | **Open source license** | **Location of the file** |
| *For example: Design file 1* | *e.g., CAD files, figures, videos* | *All designs must be submitted under an open hardware license. Enter the corresponding open source license for the file.* | *Either enter the URL for the repository or the sentence: "Available with the article".* |
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*For each design file listed in the summary table above, include a short description of the file below (just one or two sentences per design file).*

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*If your bill of materials is long or complex, you can upload the details in an editable spreadsheet, e.g., ODS file type, Excel spreadsheet or PDF file, to an open access online location, such as the* [*Open Science Framework*](https://osf.io/)*repository. Include the link here. Alternatively, the bill of materials can be submitted alongside your manuscript as supplementary material.*

1. **Bill of materials summary**

*Complete a separate row for each component of your hardware – all components associated with a cost should be listed and any empty rows should be deleted.*

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*You can use this space for any additional descriptions of the materials used.*

1. **Build instructions**

Diagram, schematic

Description automatically generated

Figure 2. Schematic of CLSLab-Liquid, including liquid and electrical connections and pinout diagram. Pico W and wire mounting of Grove LED, AS7341, and five-way manifold not shown.

1. Liquid connections
   1. Cut off the tubing just above the blue spikes
   2. Fill four of the drip bags with 1 gallon of water each
   3. Add 40, 60, and 30 food coloring drops for the red, yellow, and blue drip bags, respectively
   4. Adjust the water flow restrictor to the “off” position for each of the bags
   5. Attach the end of each of the four drip bags to one end of each of the four peristaltic pumps (note: you may need to switch which one you’re using later depending on the electrical wiring, as peristaltic pumps are reversible)
   6. Insert the end of the shorter sealing lid tube into one of the ports of the five-way manifold
   7. Connect the red, yellow, blue, and clear drip bags to the other ports of the five-way manifold
   8. The drip bags can be hung using the provided S-hooks
2. Electrical
   1. Connect the AS7341 to Grove Port #6 via the Grove to Stemma-QT adapter
   2. Connect the Grove LED Grove Port #5 via the Grove to Grove adapter and insert the white LED into the Grove LED slot. You may need to try flipping the LED to ensure the proper orientation. Note: You will also need to use a mini screwdriver to turn the potentiometer to the lowest resistance setting (highest power)
   3. Solder the stripped ends of wires (each at least 6 inches) to each of the leads on the peristaltic pumps (peristaltic\_pump), totaling eight wires. Ensure that the loose end of each wire is also stripped (~1/4”)
   4. Fashion four pieces of bare wire ~3/4” long and use these to connect the terminal block jack adapter (terminal\_block\_jack) to the terminal block leads of each of the motor drivers. Ensure that the polarities match up (+ to +, - to -)
   5. For each peristaltic pump, insert the two wire leads into a set of + and – terminal block leads on one of the motor drivers and secure the connection with a screwdriver. Note that you may need to either swap the + and – wires or swap the tubing connections to the peristaltic pump to ensure the correct direction of flow
   6. Connect the motor driver pins to the Maker Pi Pico base female headers using jumper cables according to the pinouts from Table 1:
   7. Plug the 12V wall adapter into an outlet, connect the barrel plug to the splitter cable, and then connect each of the pigtails on the ends of the splitter to the terminal block adapters connected to the Maker MDD3A modules

Table 1. Pinout table for Pico W to Maker MDD3A motor drivers. See also Figure 2. Ensure that GND is connected on each module. VBUS only needs to be connected to 5V0 on one of the Maker MDD3A modules.

|  |  |  |
| --- | --- | --- |
| Pico W | Maker MDD3A | Drip Bag |
| GP0 | M1A (first) | Red |
| GP1 | M2A (first) | Yellow |
| GND | GND (first) | - |
| GP2 | M1A (second) | Blue |
| GP3 | M2A (second) | Rinse water |
| GND | GND (second) | - |
| VBUS | 5V0 (second) | - |

1. Flowthrough Cuvette
   1. With ample water, sand off the closed end of the plastic cuvette (cuvette) using the sandpaper (sandpaper\_120), making it a both-ends open cuvette. This can be done by repetitively scraping the cuvette along the sandpaper
   2. Use the 7/32” leather hole punch (hole\_punch) to make holes in two of the sealing lids (sealing\_lid)
   3. Cut two inches of tubing from one of the drip bags, and insert it through one of the sealing lids until it is protruding ~1/8”
   4. Take the end of the tube for the fifth drip bag (the waste bag) and insert it through the other sealing lid until it is protruding ~1/8”
   5. Apply super glue to both sides of the sealing lid, being careful not to plug up the tubing, and ensuring that a good seal is formed between the exterior of the tube and the lid
   6. Once dry, apply super glue to the exterior of each sealing lid, and insert the sealing lids into either side of the cuvette.
   7. Apply super glue to the seams adjoining the sealing lid and the cuvette on either edge and wait for it to dry
   8. If available, electrical tape can be added around the seams to prevent the sealing lid from moving and breaking the hermetic super glue seal
2. Mounting
   1. Use the wire as sculpting or mounting wire by threading it through the holes on the AS7341 light sensor, and twisting the two loose ends together, then attach one of the loose wire ends to one of the mounting holes on the Maker Pi Pico base via a binding post (to enable modularity)
   2. Do the same for the Grove LED module and the five-way manifold, using separate binding posts and mounting holes on the Maker Pi Pico base. One binding post and one mounting hole can be used for each of the holes on the five-way manifold to improve the mechanical rigidity (due to it being heavier than the electrical boards)
   3. Position the Grove LED so that it is pointing perpendicular towards one face of the cuvette
   4. Position the AS7341 light sensor so that the pinhole is facing the opposite end of the cuvette, and make sure both the Grove LED and AS7341 are positioned near the middle of the rectangular face of the cuvette on either side
3. Software
   1. Flash MicroPython onto the Pico W
   2. Upload sdl\_demo\_liquid.zip source files from the latest release at <https://github.com/sparks-baird/self-driving-lab-demo/releases> to the Pico W
   3. Enter the appropriate credentials into secrets.py using the Thonny IDE / editor (<https://thonny.org>)

*Provide detailed, step-by-step construction instructions for the submitted hardware:*

* *Include all necessary information for reproducing it.*
* *Explain and (when possible) characterize design decisions. Include any design alternatives you created.*
* *Use visual instructions such as schematics, images and videos.*
* *Clearly reference design files and component parts described in the* ***Design file summary*** *and the* ***Bill of materials summary****.*
* *Highlight any potential safety concerns.*

1. **Operation instructions**
2. Run main.py using the Thonny IDE / editor
3. Note the PICO\_ID that prints to the command line when running main.py
4. Run the following notebook using the PICO\_ID from step 2: <https://github.com/sparks-baird/self-driving-lab-demo/blob/main/notebooks/3.3-random-vs-grid-vs-bayesian-liquid.ipynb>
5. Be careful to replace the waste bag before it’s full, as overfilling will lead to leaking, likely at one of the peristaltic pump connection locations
6. For larger capacity, the water and waste IV drip bags can be replaced by 5-gallon buckets with a slit cut into the lids of each to accommodate the tubing. Please follow the safety recommendations, keeping both buckets closed / away from small children to avoid a drowning hazard

*Provide detailed, step-by-step instructions for the safe and proper operation of the hardware.*

* *Use visual instructions, as necessary.*
* *Highlight any potential safety hazards.*

1. **Validation and characterization**

CLSLab-Liquid is a useful tool for teaching optimization concepts. The optimization efficiency of three different search algorithms was assessed using CLSLab-Liquid. Bayesian optimization was most efficient, followed by random search and then grid search.

Chart, line chart

Description automatically generated

Figure 3. Color mismatch (Frechet distance) vs. iteration number for grid search, random search, and Bayesian optimization efficiency of the CLSLab-Liquid color-matching task. Five search campaigns were run across each of the search types, with 27 iterations per search campaign, totaling 405 iterations.

Video of the cuvette getting rinsed and then filled again (YouTube).

*Demonstrate the operation of the hardware and characterize its performance for a specific scientific application.*

* *Highlight a relevant use case.*
* *If possible, characterize performance of the hardware over operational parameters.*
* *Create a bulleted list describing the capabilities (and limitations) of the hardware. For example, load and operation time, spin speed, coefficient of variation, accuracy, precision, etc.*

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*Funding: This work was supported by the National Institutes of Health [grant numbers xxxx, yyyy]; the Bill & Melinda Gates Foundation, Seattle, WA [grant number zzzz]; and the United States Institutes of Peace [grant number aaaa].*

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*This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.*

**References:**

*If relevant, you should**include a reference to the original publication of the hardware you customized and a reference to the repository in which your design files are published. Other references can be included, as required; for example, references that put your device in context in the literature. For more information on the reference format in HardwareX please see the* [*Guide for Authors*](https://www.elsevier.com/journals/hardwarex/2468-0672/guide-for-authors)*.*

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***Author manuscript checklist***

* *Is the subject of the submission under an open source license? Are design files in the preferred format for making modifications as defined by the* [*Open Source Hardware definition*](http://www.oshwa.org/definition/)*?*
* *Can the hardware be reproduced with the details provided in the submission?*
* *Are all relevant design files available on either the Mendeley Data, Open Science Framework, or Zenodo repository? Are they described in the Design Files Summary, and clearly documented? (E.g., descriptive file names, commented code, labeled images, etc.)* 
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* *Are visual instructions used when necessary?*
* *Is the utility of the hardware to the scientific community explained clearly? Has a specific scientific application been demonstrated using the hardware?*
* *Is the performance of the hardware adequately demonstrated and characterized?*
* *Are all potential safety concerns addressed?*
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