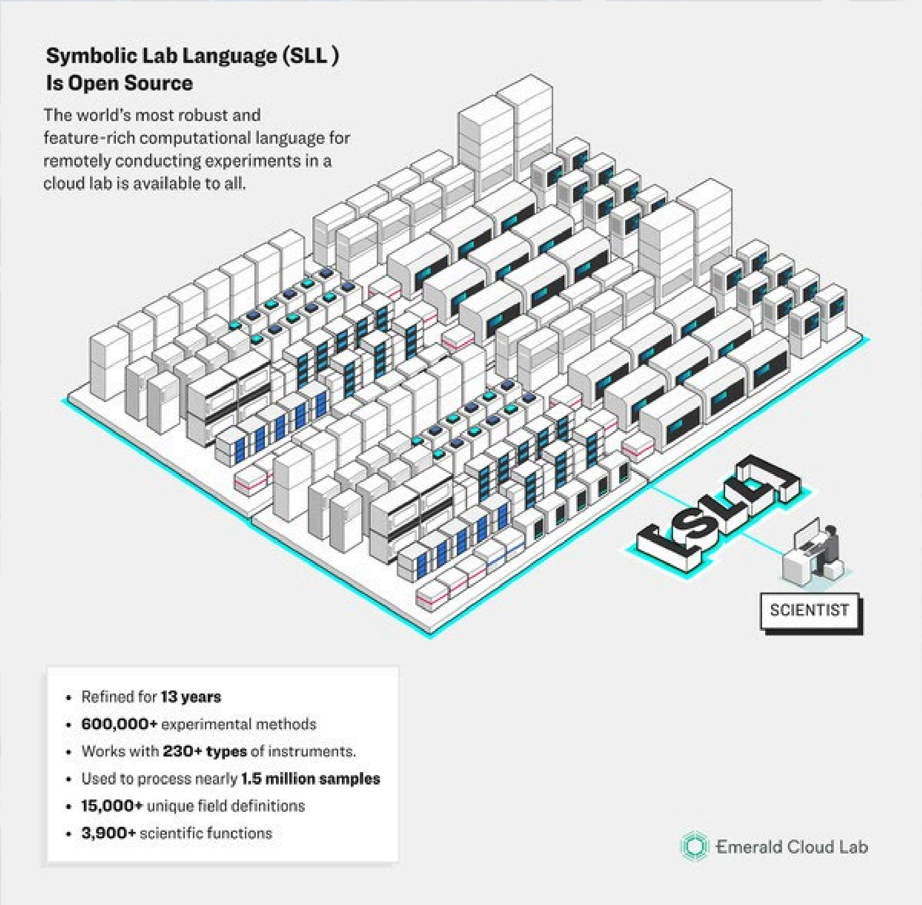
Closed-loop and Autonomous: CMU Cloud Lab for Measuring Hansen Solubility Parameters



Sijie Fu

Thursday, June 27, 2024

Washburn Group @ CMU

covestro

ECL

Carnegie Mellon University Cloud Lab

Carnegie Mellon University

Outline

intro

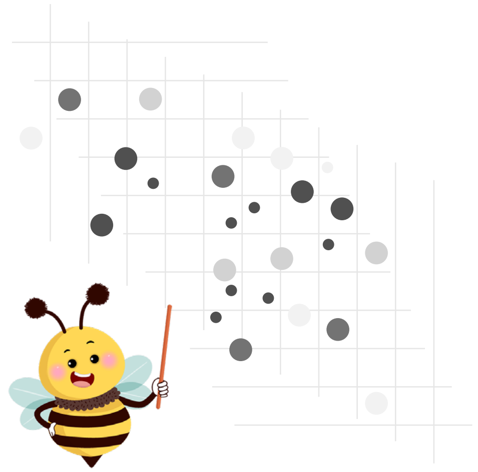
Introduction

- The basics of the CMU cloud lab
- Hansen Solubility Parameters (HSPs)

aim

Inspiration

- Why the cloud lab? Why ***autonomous***?



the cloud lab
worker bee

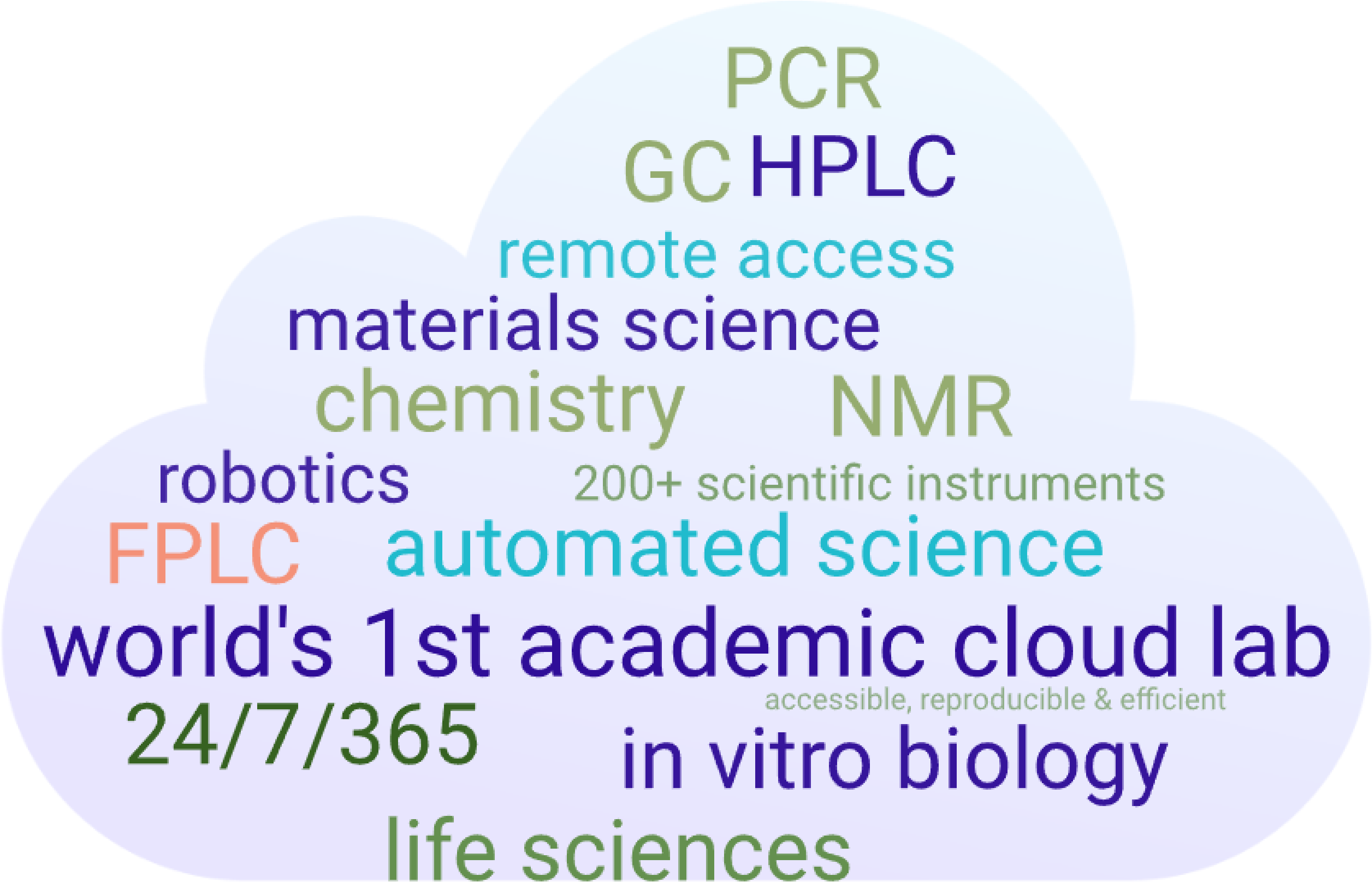
Some interesting demos

method

Approach

- The closed-loop cloud lab ***workflow***
- Computer vision (CV) for automated experiment ***result analysis***
- A Bayesian-based machine learning (ML) model for planning and ***selecting experiments***

Basics of the CMU cloud lab



Symbolic Lab Language (SLL)

the "heart" of the cloud lab

1. find a 100 mL volumetric flask & label it

2. transfer 5g of NaCl into the container (*solid transfer protocol*)

3. fill to volume with water

```
(* A code snippet to make a 5% NaCl solution in the cloud lab *)
ExperimentSamplePreparation[
    {
        LabelContainer[
            Label -> "ChemSeminar: 5% NaCl",
            Container -> Model[Container, Vessel, VolumetricFlask, "100 mL Glass Volumetric Flask"]
        ],
        Transfer[
            Source -> Model[Sample, "Sodium Chloride"],
            Destination -> "ChemSeminar: 5% NaCl",
            Amount -> (5 * Gram)
        ],
        FillToVolume[
            Sample -> "ChemSeminar: 5% NaCl",
            TotalVolume -> (100 * Milliliter),
            Solvent -> Model[Sample, "Milli-Q water"]
        ]
    }
]
```

A code snippet to make a 5% NaCl solution in the cloud lab intro 1

SLL is all you need (?)

```
(* A code snippet to make a 5% NaCl solution in the cloud lab *)
ExperimentSamplePreparation[
    {
        LabelContainer[
            Label -> "ChemSeminar: 5% NaCl",
            Container -> Model[Container, Vessel, VolumetricFlask, "100 mL Glass Volumetric Flask"]
        ],
        Transfer[
            Source -> Model[Sample, "Sodium Chloride"],
            Destination -> "ChemSeminar: 5% NaCl",
            Amount -> (5 * Gram)
        ],
        FillToVolume[
            Sample -> "ChemSeminar: 5% NaCl",
            TotalVolume -> (100 * Milliliter),
            Solvent -> Model[Sample, "Milli-Q water"]
        ]
    }
]
```

- ExperimentSamplePreparation
- ExperimentDNASynthesis
- ExperimentTotalProteinDetection
- ExperimentHPLC
- ExperimentNMR
- ExperimentMeasureViscosity
- *and more…*

https://www.emeraldcloudlab.com/documentation/

Hansen solubility parameters (HSPs)

like dissolves like | *like seeks like*

δD: Dispersion (*van der Waals*)

δP: Polarity (dipole moment)

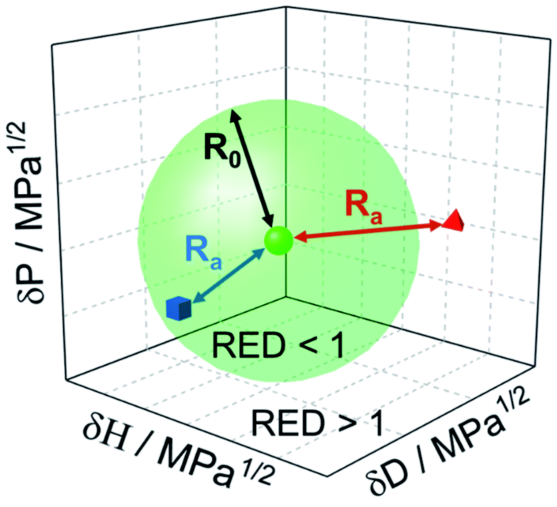
δH: Hydrogen bonding

every material can be represented with a set of $(\boldsymbol{\delta D}, \boldsymbol{\delta P}, \boldsymbol{\delta H})$ **coordinates** in the Hansen space

$$R_a^2 = 4(\delta D_1 - \delta D_2)^2 + (\delta P_1 - \delta P_2)^2 + (\delta H_1 - \delta H_2)^2$$

Relative energy difference: $RED = \frac{R_a}{R_o}$

$RED < 1.0$: *miscible;* $RED > 1.0$: *immiscible*



the Hansen space

every material has a "miscible" sphere

[Hansen Solubility Parameters | Hansen Solubility Parameters (hansen-solubility.com)](hansen-solubility.com)

Bapat, S.; O. Kilian, S.; Wiggers, H.; Segets, D. Towards a Framework for Evaluating and Reporting Hansen Solubility Parameters: Applications to Particle Dispersions. *Nanoscale Advances* **2021**, *3* (15), 4400–4410.

intro 2

HSP application: solvent optimization

coatings | filled polymer systems | …

δD: Dispersion (*van der Waals*)

δP: Polarity (dipole moment)

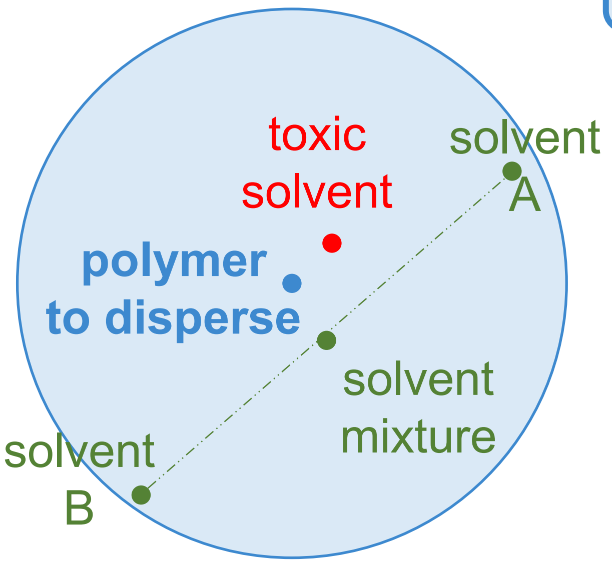
δH: Hydrogen bonding

the HSPs of a **mixture solvent** is the **linear combination** of the HSPs of it **constituting solvents** by their volume fractions

$$\delta D' = c_1 \delta D_1 + c_2 \delta D_2$$

$$\delta P' = c_1 \delta P_1 + c_2 \delta P_2$$

$$\delta H' = c_1 \delta H_1 + c_2 \delta H_2$$



a simplified 2D representation of the Hansen space

Hansen Solubility Parameters | Hansen Solubility Parameters (hansen-solubility.com)

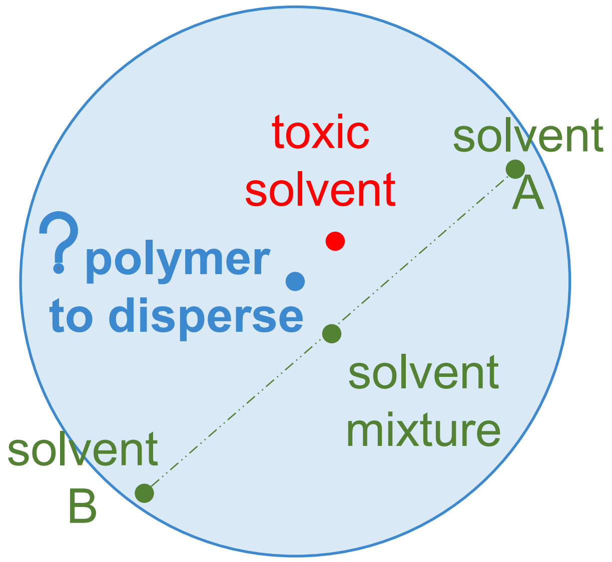
Bapat, S.; O. Kilian, S.; Wiggers, H.; Segets, D. Towards a Framework for Evaluating and Reporting Hansen Solubility Parameters: Applications to Particle Dispersions. *Nanoscale Advances* **2021**, *3* (15), 4400–4410.

How to assign HSPs?

δD: Dispersion (*van der Waals*)

δP: Polarity (dipole moment)

δH: Hydrogen bonding



theoretical molecular simulation

- ✓ alkanes should have a $\boldsymbol{\delta P}$ of 0
- ✓ water should have a high $\boldsymbol{\delta H}$
- ✓ …

but what about polymers and nanoparticles?

[Hansen Solubility Parameters | Hansen Solubility Parameters (hansen-solubility.com)](hansen-solubility.com)

HSPs: experimental measurement

where molecular simulations fail to deliver, e.g., polymers

every material has a "miscible" sphere

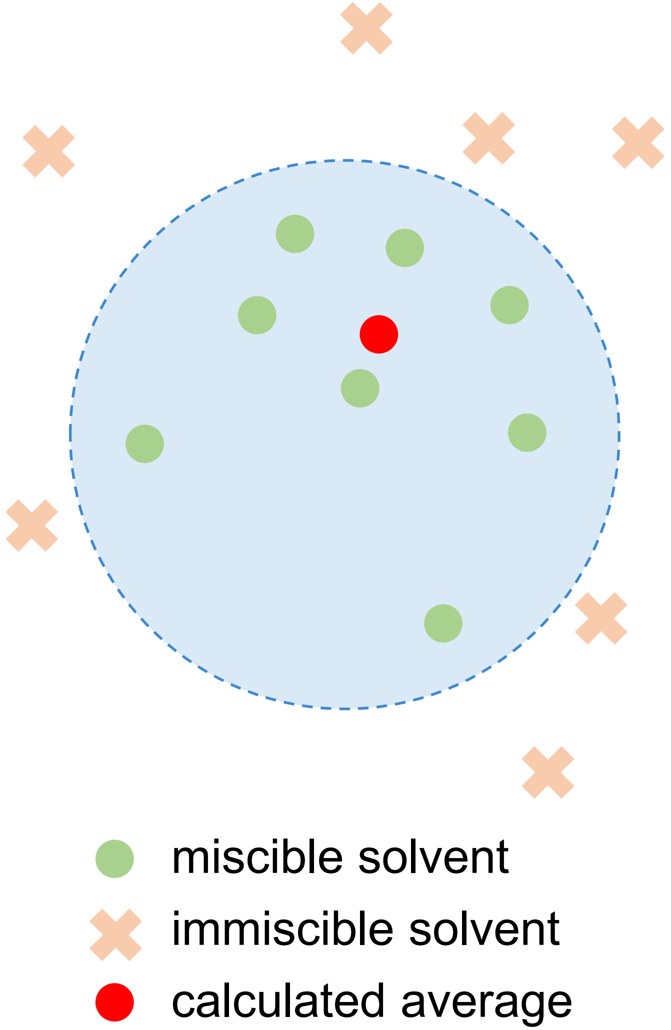
Solvents	dD	dP	dH	MVol	Score
Acetonitrile	15.3	18	6.1	52.9	0
Benzene	18.4	0	2	52.9	0
Cyclohexane	16.8	0	0.2	108.9	0
Diethyl Ether	14.5	2.9	4.6	104.7	0
1,4-Dioxane	17.5	1.8	9	85.7	0
Ethanol	15.8	8.8	19.4	58.6	0
Hexane	14.9	0	0	131.4	0
Iso-Propyl Ether	15.1	3.2	3.2	141.8	0
Methanol	14.7	12.3	22.3	40.6	0
Toluene	18	1.4	2	106.6	0
Xylene	17.6	1	3.1	123.9	0
Acetone	15.5	10.4	7	73.8	1
Chloroform	17.8	3.1	5.7	80.5	1
m-Cresol	18.5	6.5	13.7	105	1
Dimethyl Sulfoxide (DMSO)	18.4	16.4	10.2	71.3	1
1,3-Dioxolane	18.1	6.6	9.3	69.9	1
Ethyl Acetate	15.8	5.3	7.2	98.6	1
Methyl Ethyl Ketone (MEK)	16	9	5.1	90.2	1
N-Methyl-2-Pyrrolidone (NMP)	18	12.3	7.2	96.6	1
Methylene Chloride	17	7.3	7.1	64.4	1
N,N-Dimethyl Formamide (DMF)	17.4	13.7	11.3	77.4	1
Tetrahydrofuran (THF)	16.8	5.7	8	81.9	1

Gather a list of solvents w. known HSP values

Test for various solvent miscibilities

*Find **miscible** solvents*

*Calculate **average***



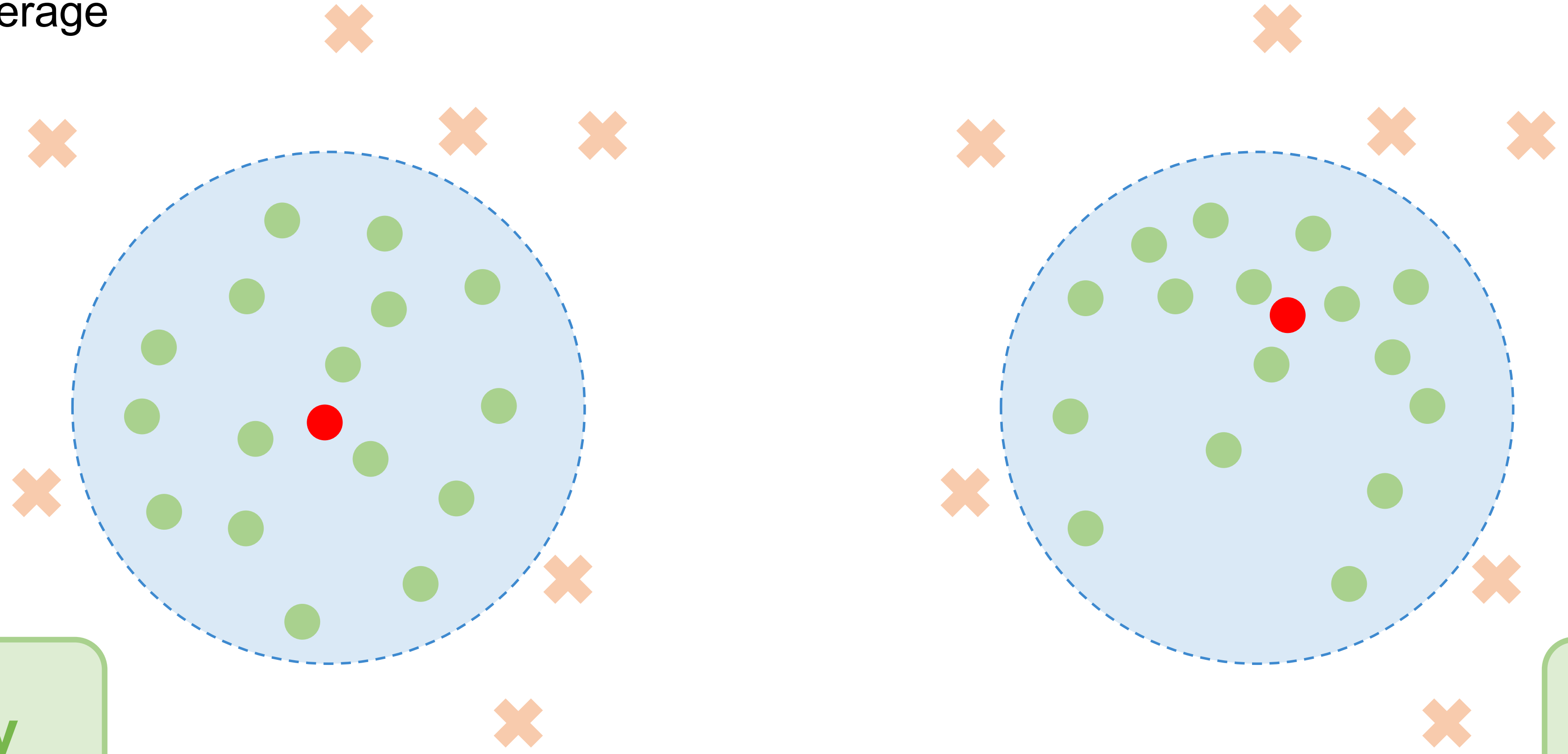
Hansen Solubility Parameters | Hansen Solubility Parameters (hansen-solubility.com)

aim

Why the cloud lab?

Automation – “free” grad students from monotonous work
🤖: easy there, not trying to take your job

- miscible solvent
- immiscible solvent
- calculated average



Ideally

Reality

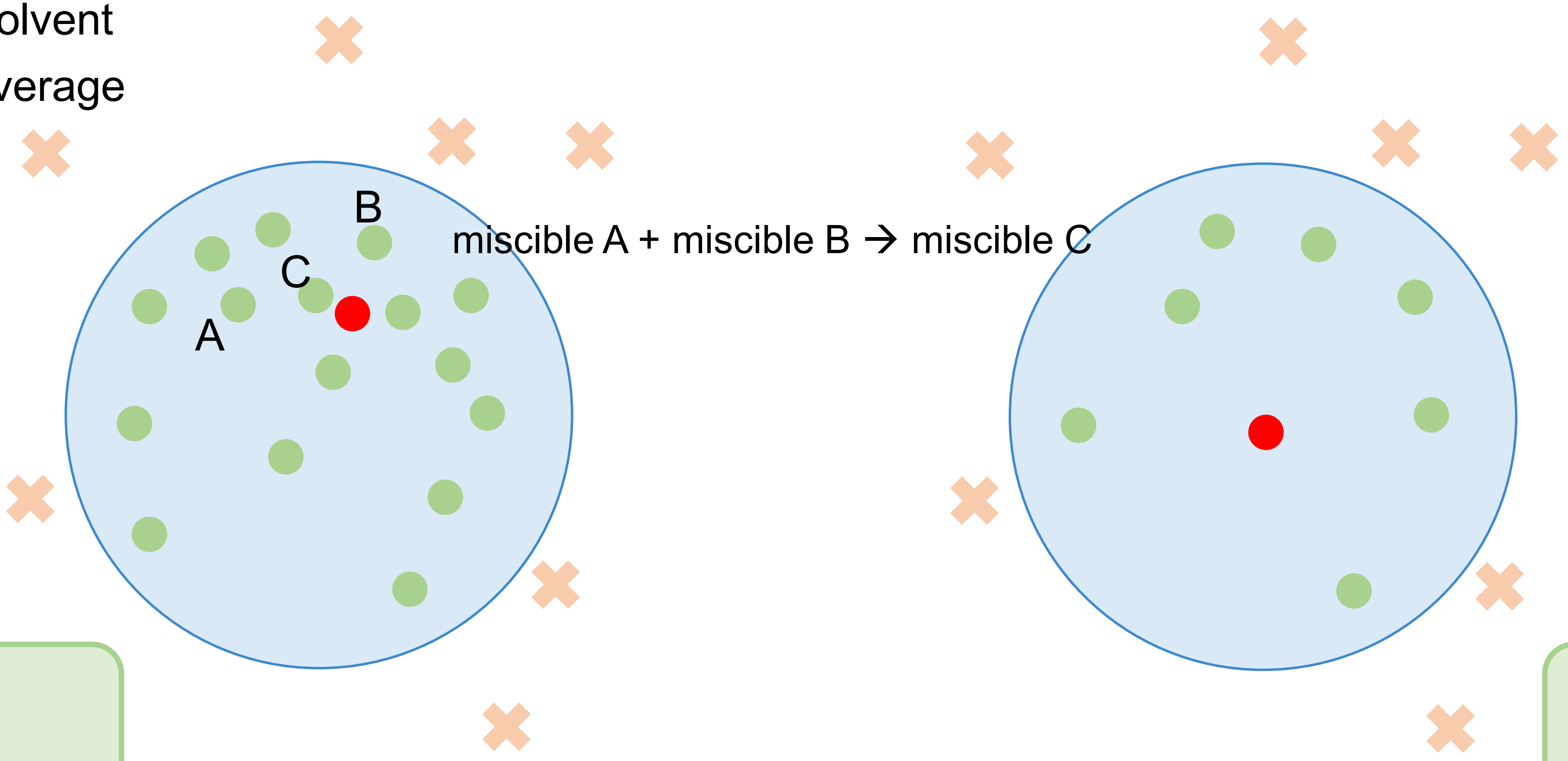
Solvent choice matters – estimation by average can be biased

aim

The more the merrier? Less could be more!

*find the **sphere center** rather than the average*

- miscible solvent
- immiscible solvent
- calculated average



Aim

Better yet

sphere-wise interpolation of the decision boundary

only “support vectors” matter

aim

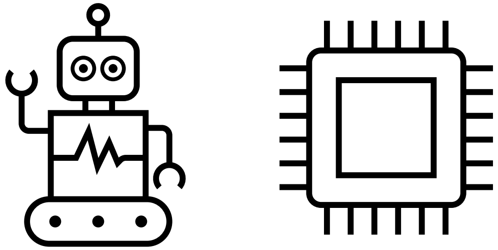
: Choose your experiments wisely!

similar solvents tend to give similar miscibility

Closed-loop & Autonomous

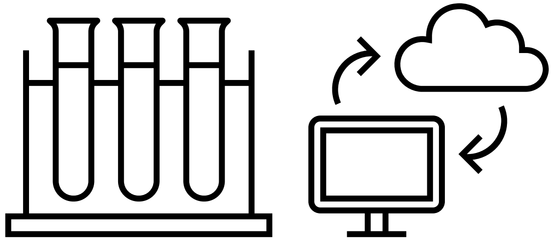
with minimal grad student input

Artificial intelligence



efficiently plan our **experiment road map** *(updated every time a new result is acquired)*

Cloud lab



remotely execute the **experiment protocols** and provide findings

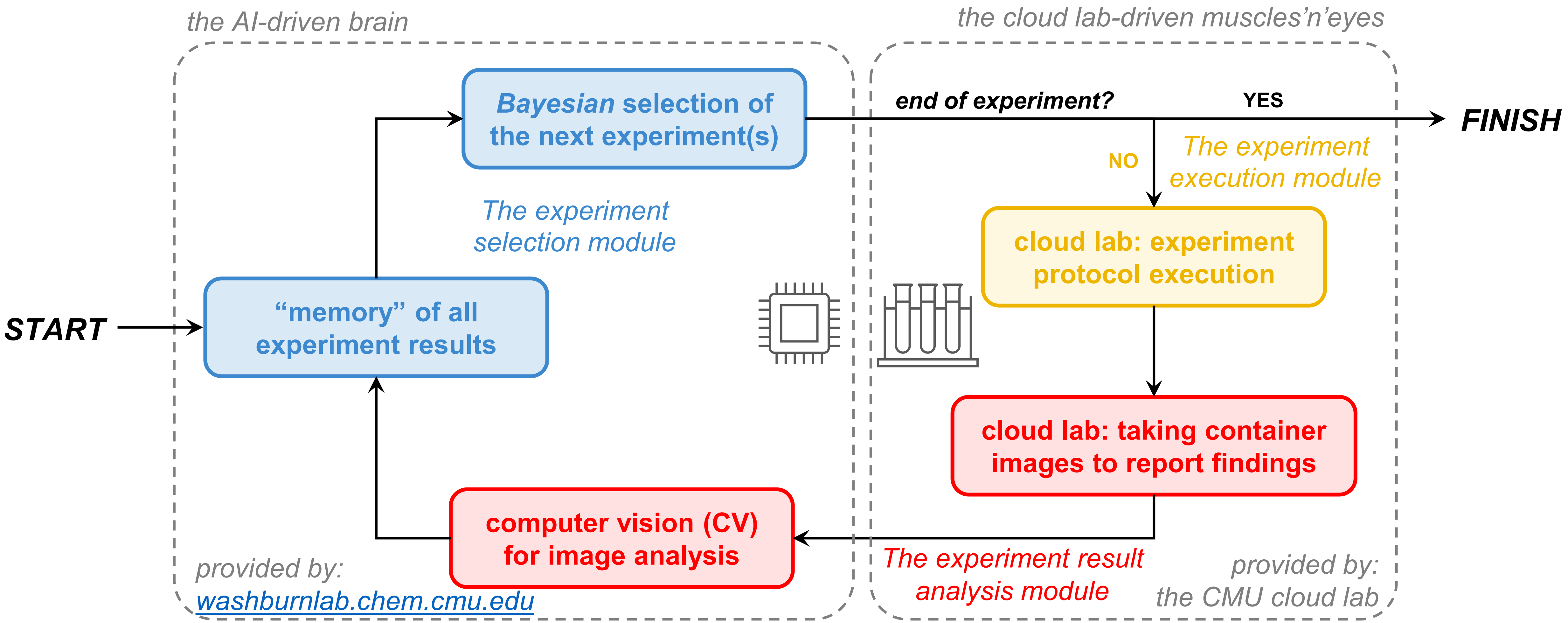
Goal

Efficiently determine the ***"miscible" sphere*** for the test material(s) with the least number of experiments (solvents) method

2 parts
3 modules

Closed-loop & Autonomous

measuring HSPs in the cloud lab with a click of a button

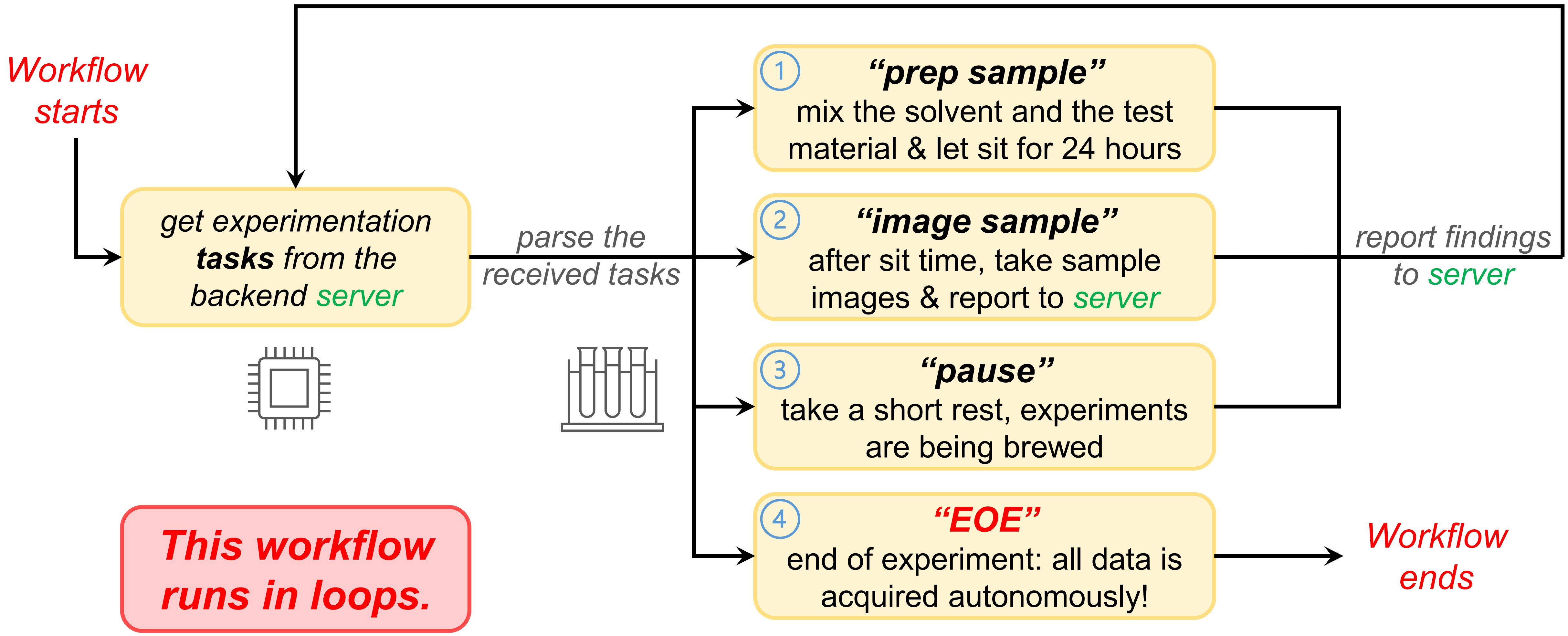


→ *Arrows represent the direction of information flow.*

method 1

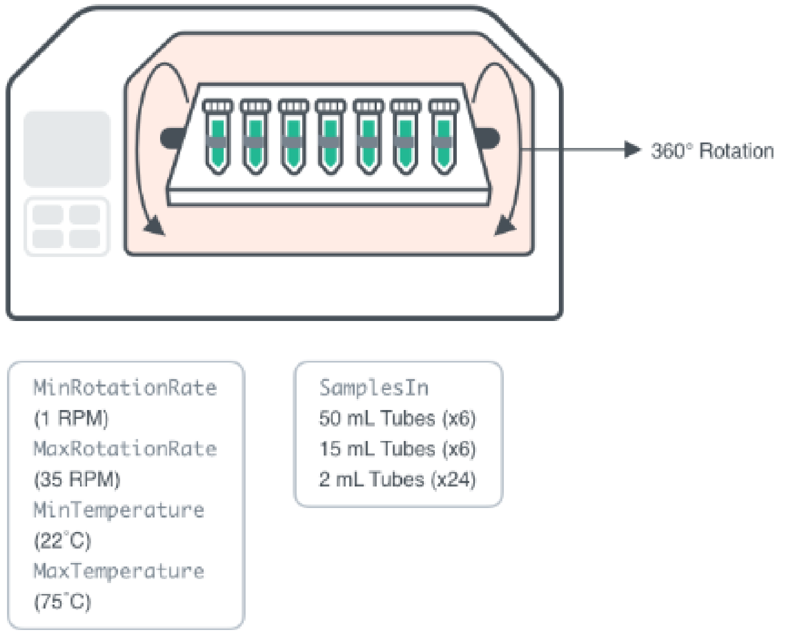
The closed-loop cloud lab workflow

the experiment execution module + result collection

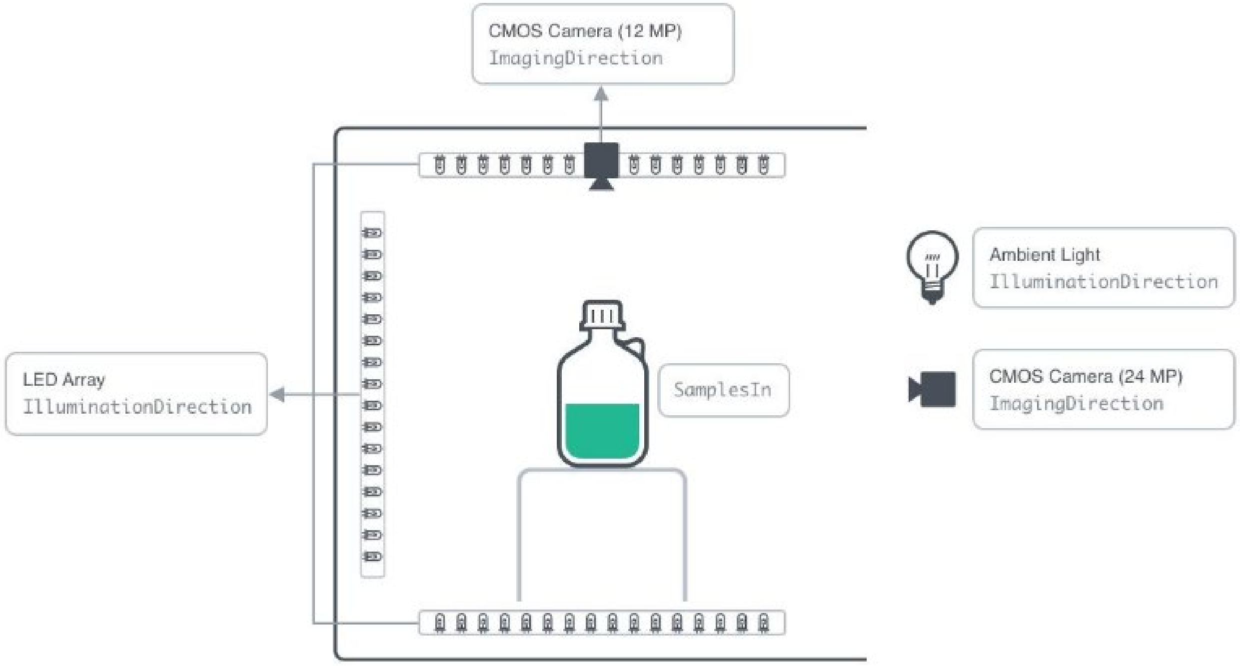


method 1

The two major cloud lab protocols



Enviro-Genie shaker for mixing
(prep sample)



camera for image collection/inspection
(image samples)

ECL Carnegie Mellon University Cloud Lab

method 1

Preliminary runs in the CMU cloud lab

5 solvents
1 test resin

miscible

miscible

immiscible

immiscible

miscible

Carnegie Mellon University
Cloud Lab

Closed-loop & Autonomous

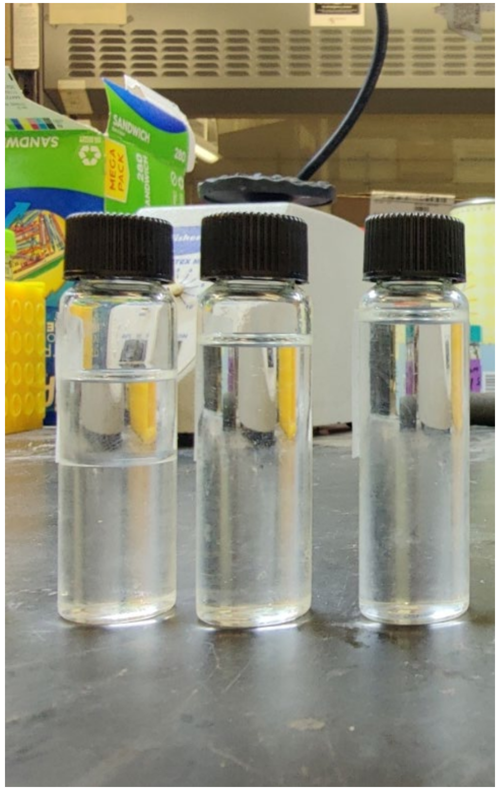
with minimal grad student input

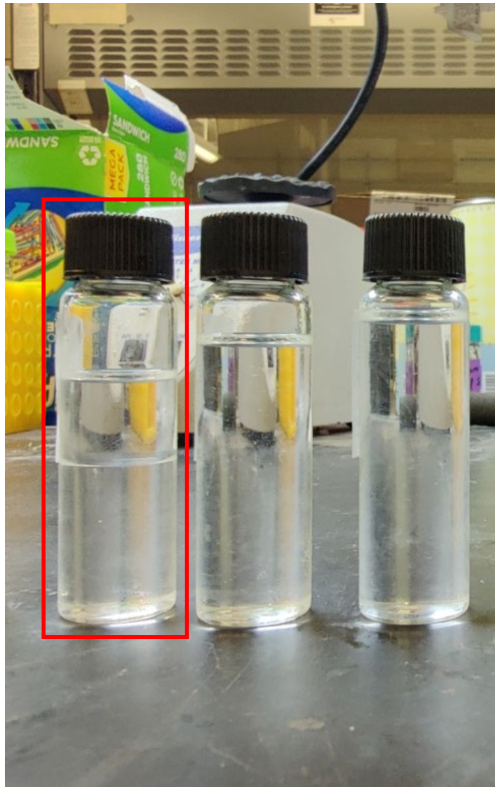
How?

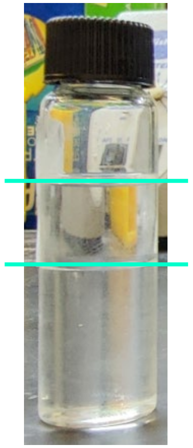
method 2

Computer vision (CV) for interface detection

Keep in mind that computers have no visual sensations – they only “see” numbers.







N

Take container image

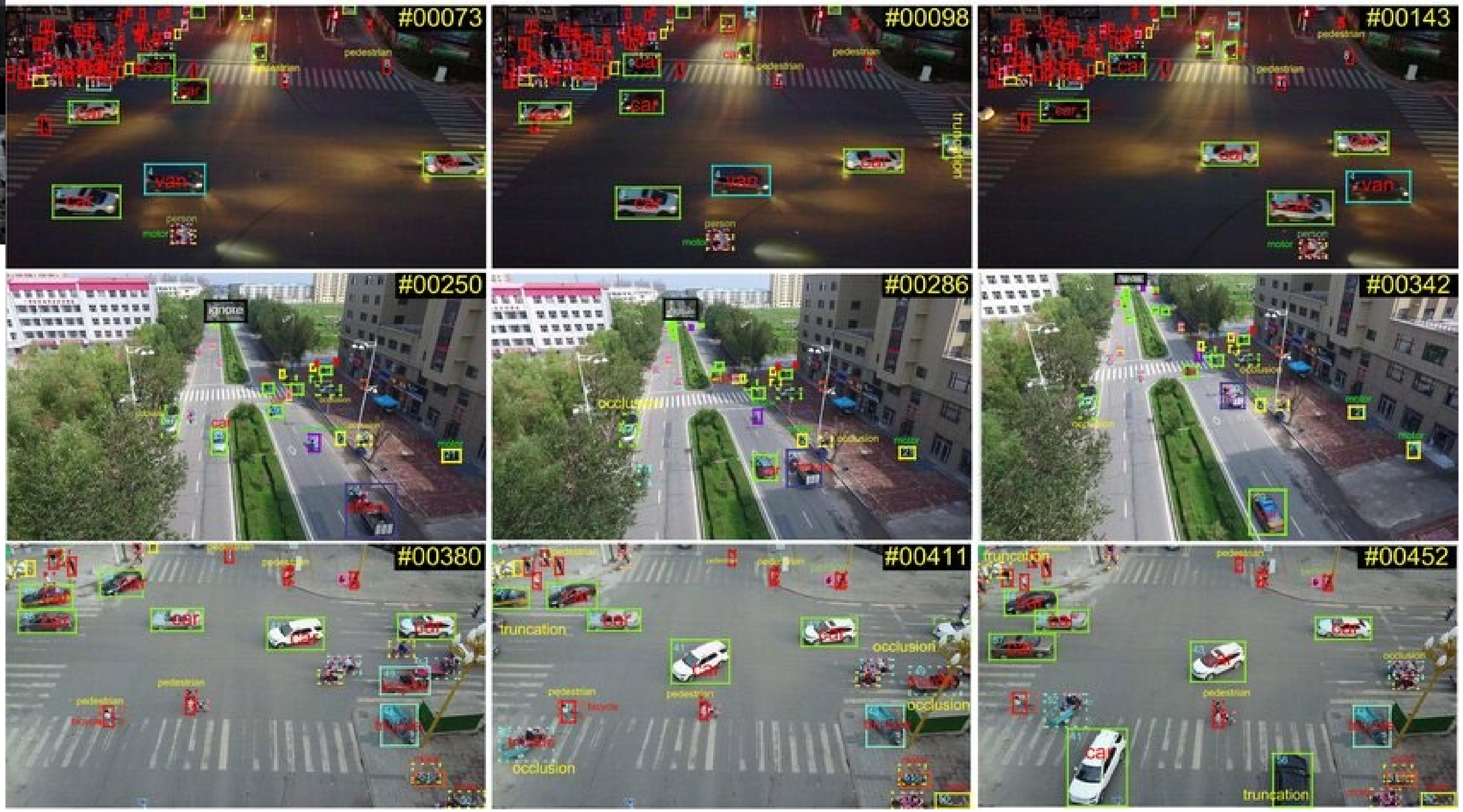
Step 1: Find vial(s) & crop image

Step 2: Locate interface(s) & make a conclusion

method 2

Object detection

Step 1: Find vial(s) & crop image

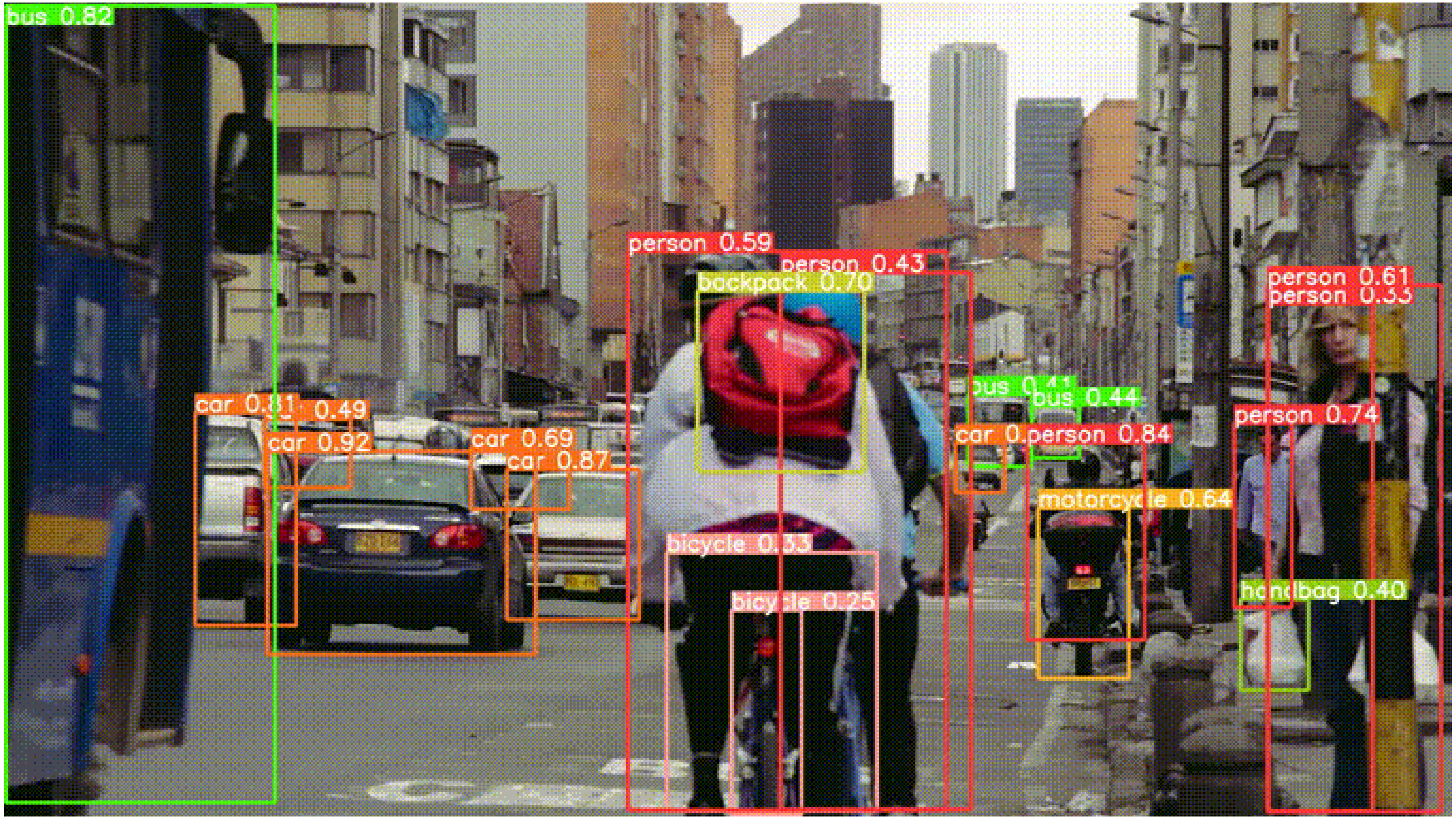


VisDrone-VDT2018: The Vision Meets Drone Video Detection and Tracking Challenge Results: Munich, Germany, September 8-14, 2018, Proceedings, Part V. 10.1007/978-3-030-11021-5_29.

method 2

Object detection: a common CV task

Step 1: Find vial(s)
& crop image



Original video: [Camilo Calderón - Photography (pexels.com)]
Annotated gif: [Enhanced Object Detection: How To Effectively Implement YOLOv8 | by Thomas A Dorfer | Towards Data Science]

method 2

Vial detection by YOLOv10n

You Only Look Once (YOLO)

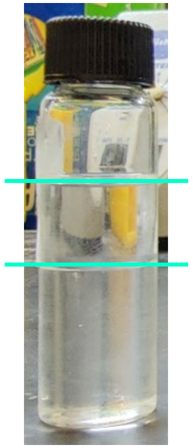
1. Find images with vials and label them;
2. Use pretrained YOLOv10n weights and ***fine-tune*** the model for vial detection.

Vial detection demo

- YOLOv10 - Ultralytics YOLO Docs
- Wang, A.; Chen, H.; Liu, L.; Chen, K.; Lin, Z.; Han, J.; Ding, G. YOLOv10: Real-Time End-to-End Object Detection. *arXiv* May 23, 2024.

method 2

Interface detection



N

Look for interface(s) &
make a conclusion

Sadly to the best of my knowledge, ***AI may not be the best option*** for such a nuanced/transparent task here, especially with the ***lack of training data***.

"*ab initio*" image analysis

edge detection

method 2

Edge detection for interface detection

horizontal edge detection : $\begin{bmatrix} 1 & 1 & 1 \\ 0 & 0 & 0 \\ -1 & -1 & -1 \end{bmatrix}$

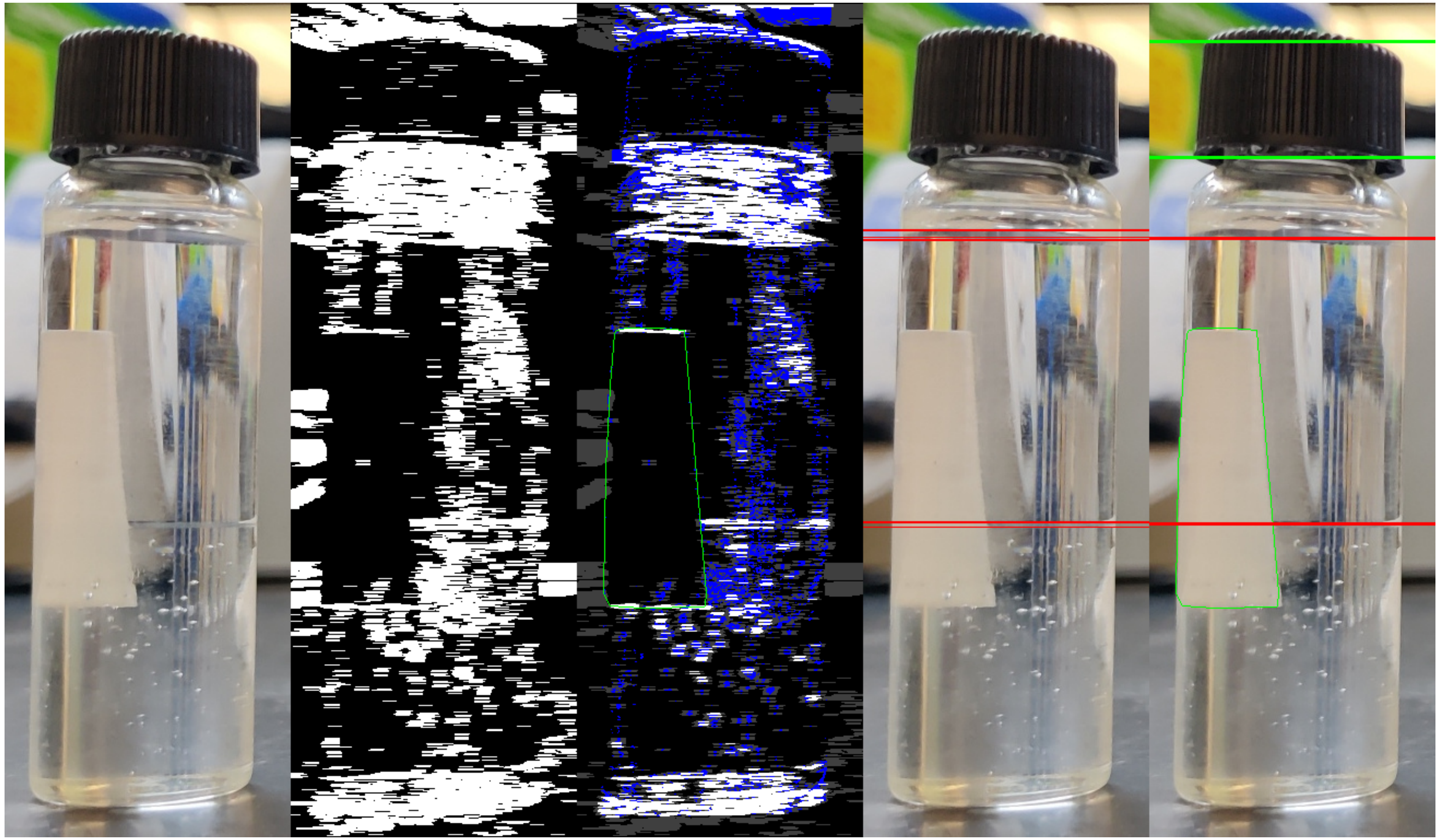
vertical edge detection : $\begin{bmatrix} -1 & 0 & 1 \\ -1 & 0 & 1 \\ -1 & 0 & 1 \end{bmatrix}$

just one simple trick made everything so much clearer!

method 2

Noise reduction, background cancelation, label correction…

- noise
- reflection
- label
- point of view
- background
- exceptions…



washburnlab.chem.cmu.edu/HSP

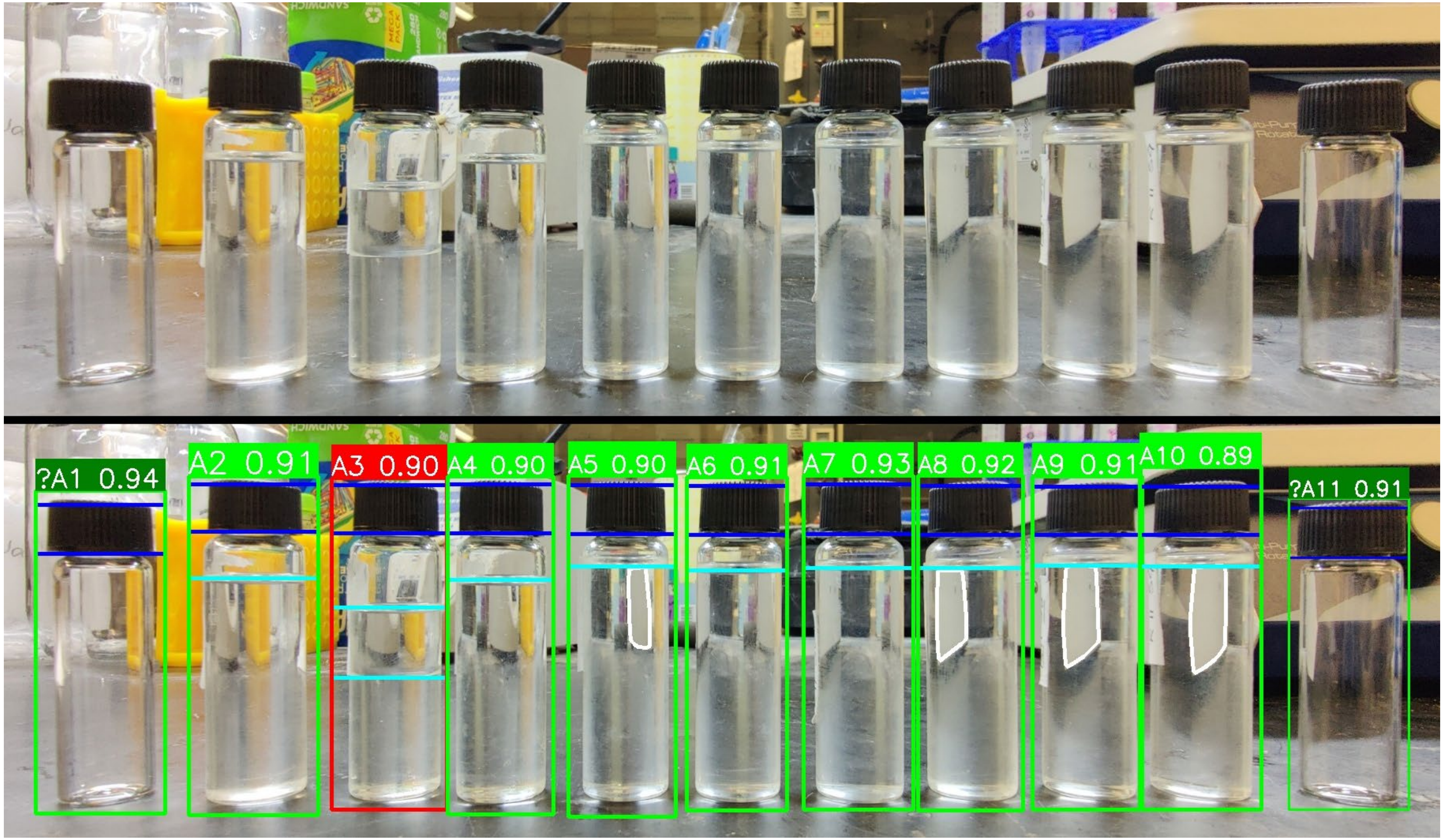
Looking for a live demo? Click the link on the left and log in. Pick any image from this search result, click on it, and then right click to “copy image link” (not the link for the whole page). Paste the image link to the webapp and have fun exploring.

method 2

Vial detection + interface detection

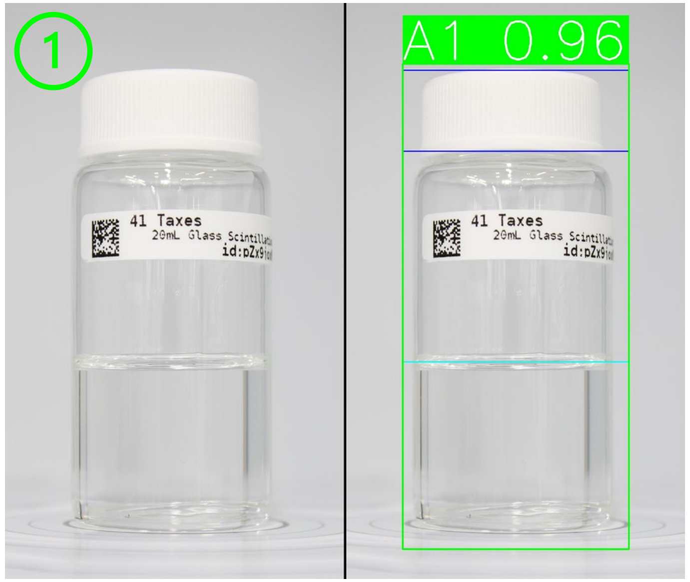
benchmarking tests

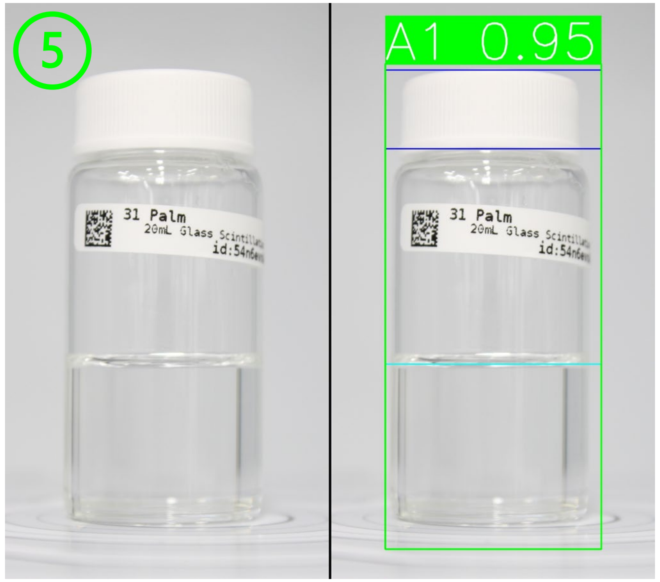
and more improvements are on the way…



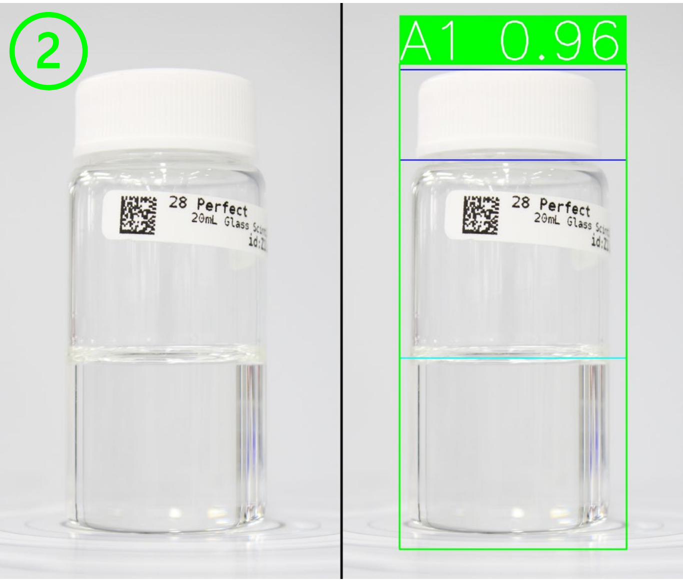
method 2

Interface detection in live action





green box:
miscible
red box:
immiscible
blue line:
cap
cyan line:
interface



100% hit rate

method 3

A Bayesian approach for planning experiments

miscible solvent

immiscible solvent

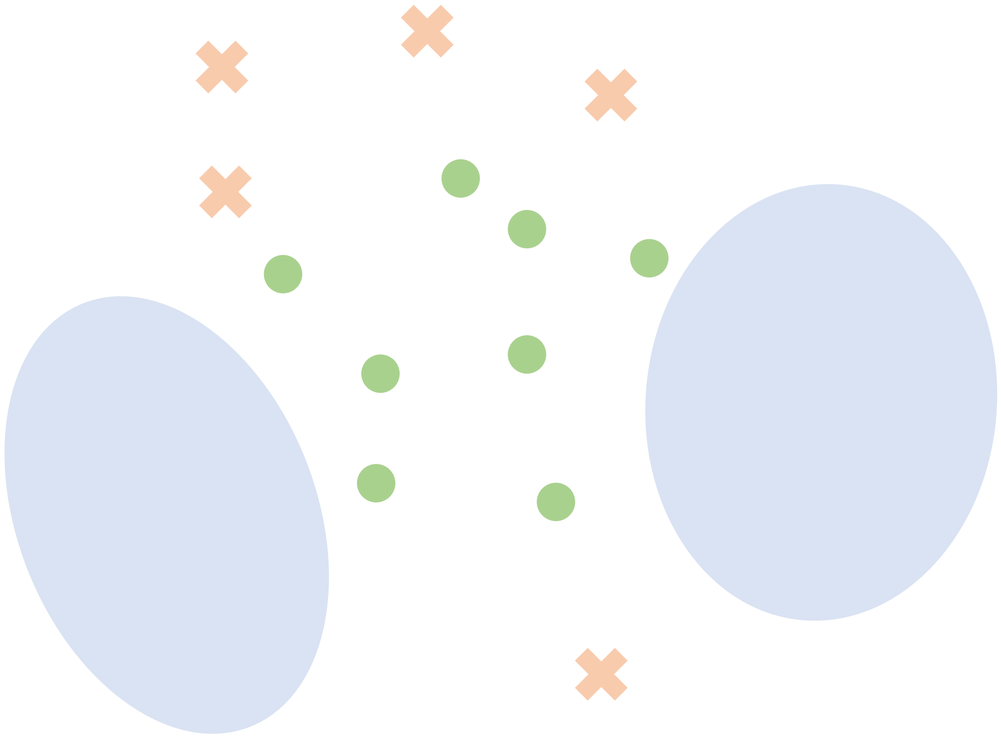
solvent selection for efficient experimentation

Principle: always prioritize experiments with ***higher uncertainties*** to maximize the ***information gain***.

helps sketch the decision boundary

explore

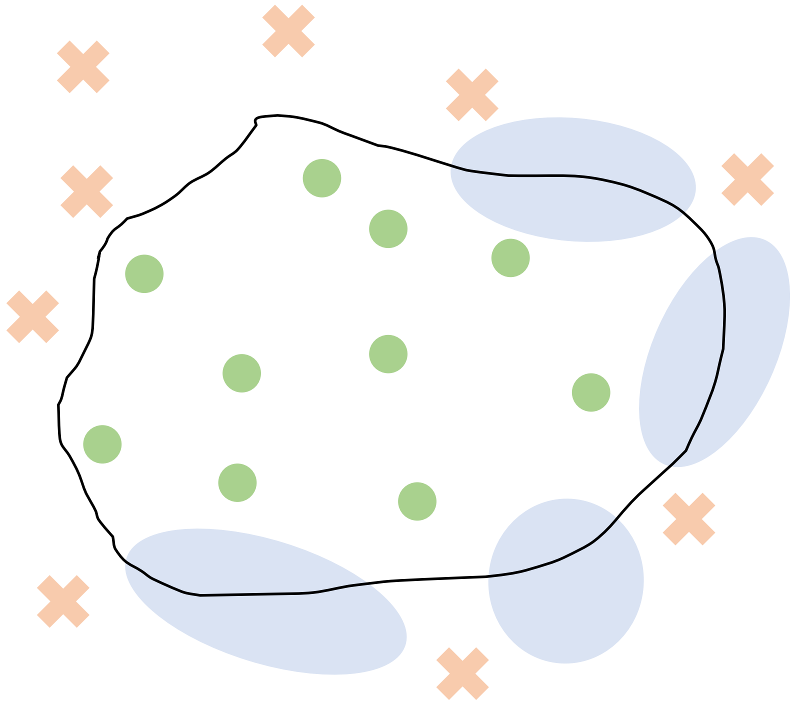
when there is *limited* knowledge, explore the regions that are ***less explored***



exploit

helps refine the decision boundary

when there is *adequate* knowledge, explore around the estimated ***decision boundary***



demo

Putting everything together

how does one closed-loop test look like?

Chemistry Summer Seminar Demo

https://washburnlab.chem.cmu.edu/HSP/

https://washburnlab.chem.cmu.edu/chem-seminar-demo/

Acknowledgements

Error loading
washburn-group.jpg

Are you sure this file exists?

Actually, I don't think we have a concurrent one yet…

ECL

Carnegie Mellon University
Cloud Lab

covestro

Prof. Newell Washburn
Prof. David Yaron
Prof. Olexandr Isayev

Amira Alakhdar
Calvin Gang

the CMU cloud lab staff

The Tilton group @ ChemE
The Garoff group @ ChemE

Lorna Williams-Rolley
Jessica Derenburger
Tina Lin and the summer seminar committee

all of you and more!

Thank you for your attention!

Happy to take any questions.

Sijie Fu

Thursday, June 27, 2024

Washburn Group @ CMU

Carnegie Mellon University