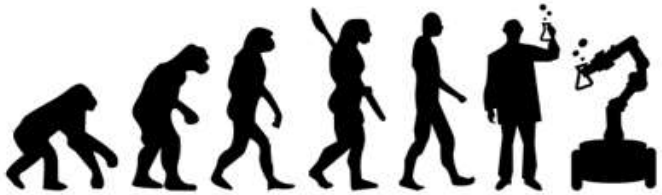
Catalysis Hub - Swiss CAT+



Automation in Analytical chemistry:
Current state and Challenges

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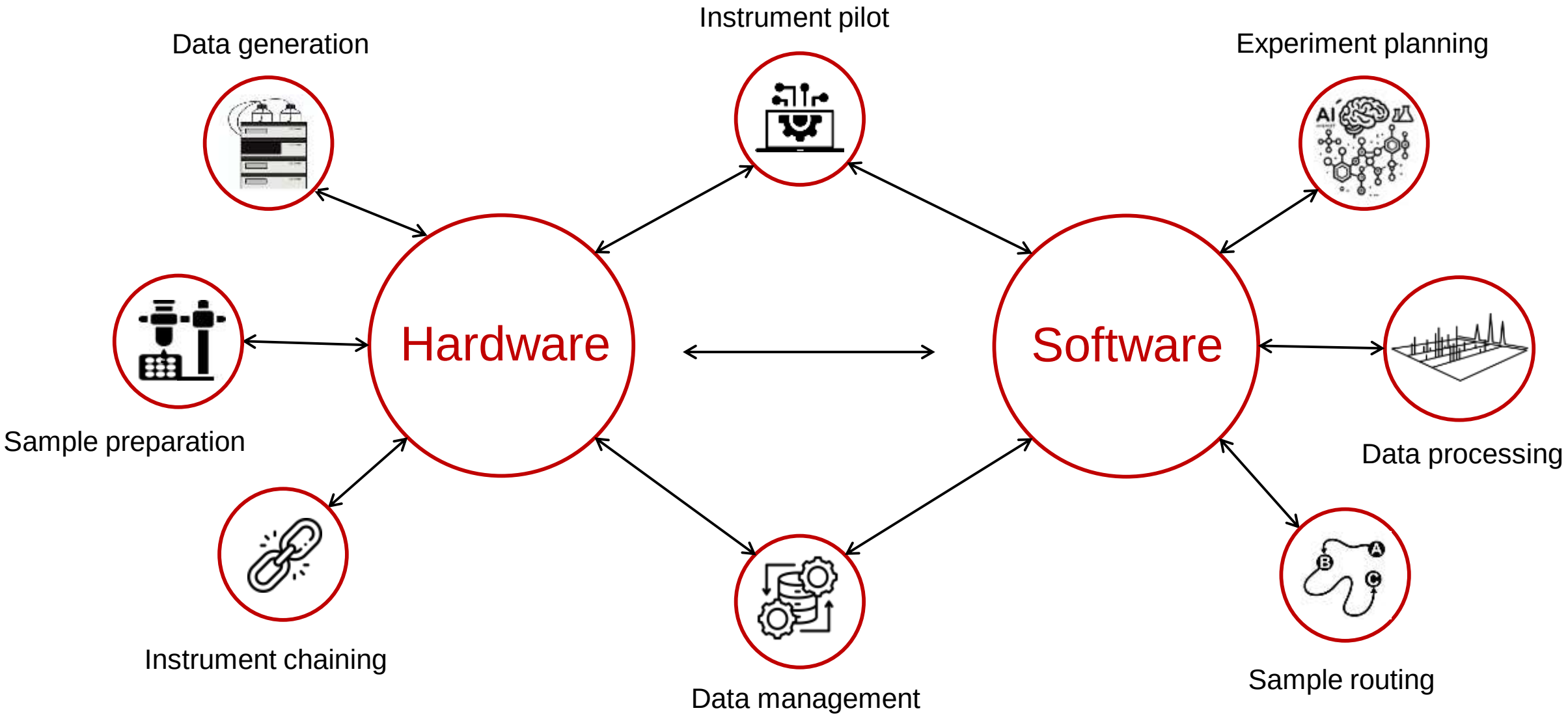
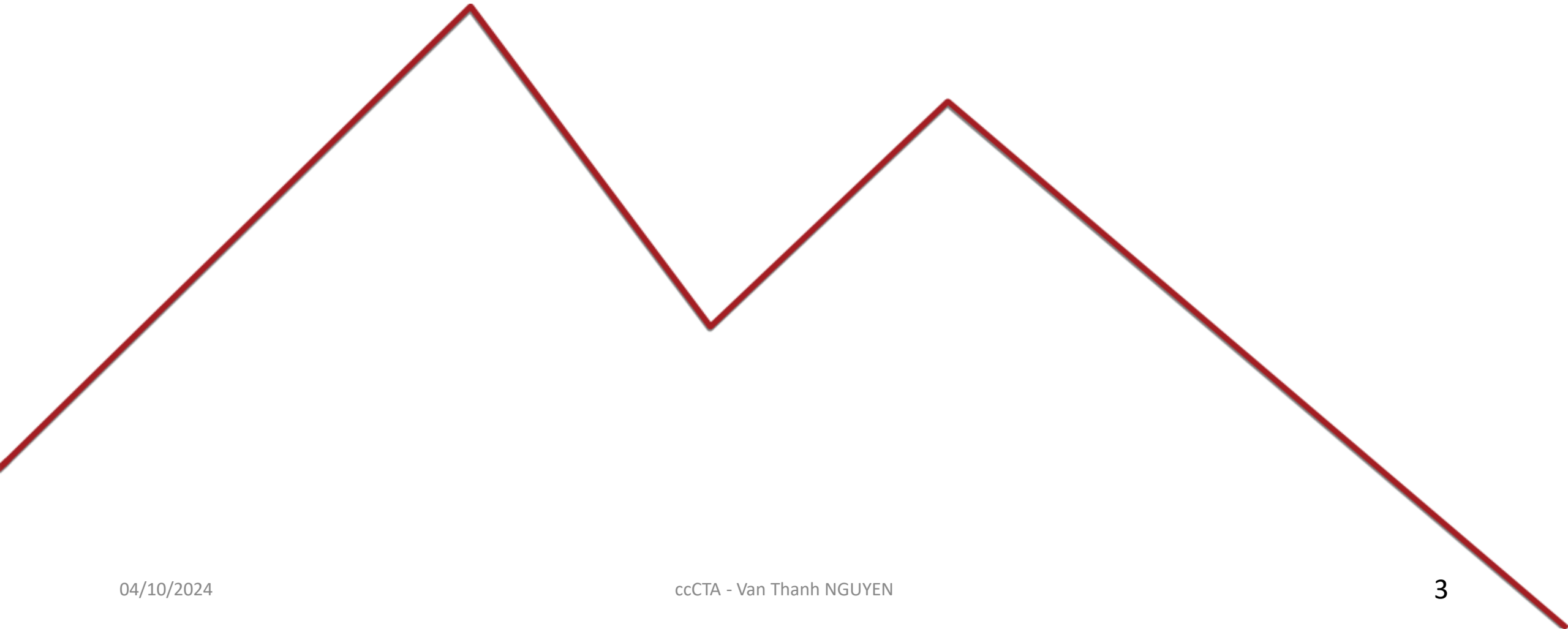
Postdoc - Collaboratrice scientifique

Villars-sur-Ollon
04 Octobre 2024

Automation in Analytical Chemistry



Hardware



SYNTHESIS SAMPLE

HPLC-DAD-ELSD-MS (SQ)

SAMPLE SELECTION ALGORITHM

Known

YIELD CALCULATION

CHIRAL

DAD FRACTION COLLECTION

EE CALCULATION

SAMPLE PREP

SFC-DAD-MS(SQ)

Screening

Unknown

SELECTED SAMPLES

Semi prepLC-DAD-MS

DAD FRACTION COLLECTION

SAMPLE PREP

OMNIFIRE

Dissolve CDCl3/MeOD

Dillution

IM-QTOF

HR-UV

FTIR

NMR

Characterization

Screening

1 SFC-DAD-SQ

2 LC-DAD-SQ-FC/ELSD

1 GC-MS

High-throughput screening

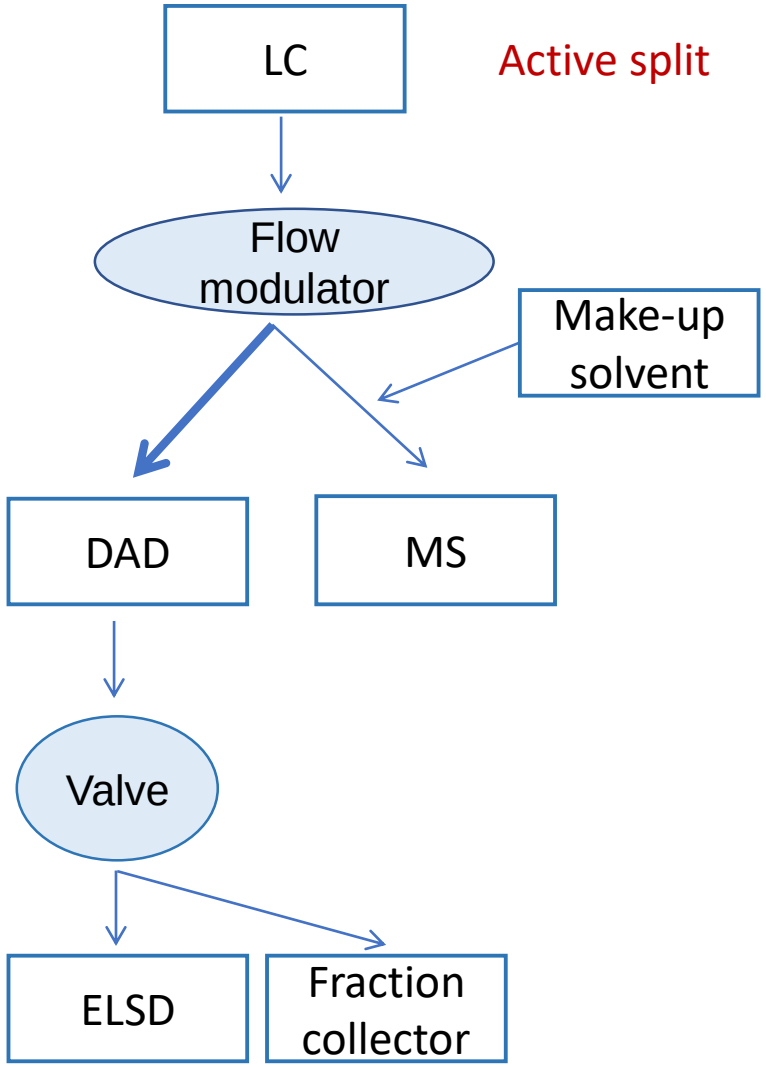
- Fast and accurate
- Robust analytical methods
- Wide range of detection for diverse molecules
- Price

➔ 2 LC systems, 1 GC

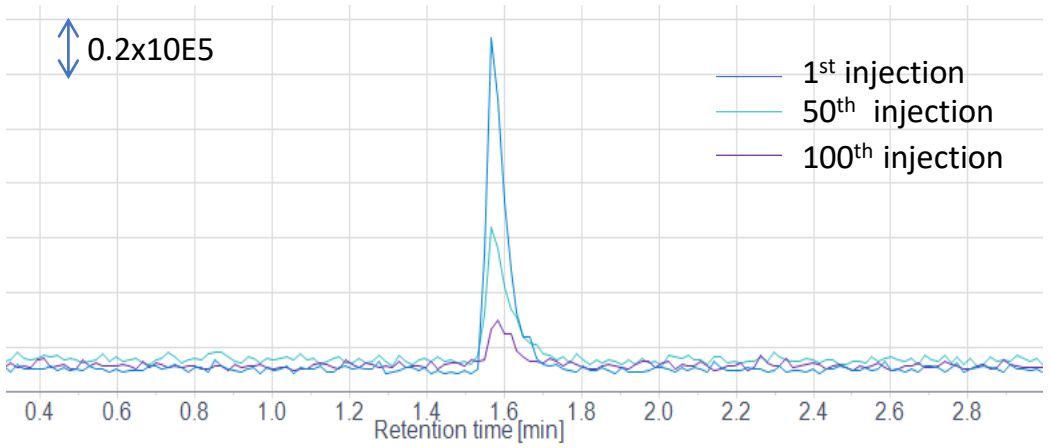
➔ Kits of 4 columns

➔ 3 detectors ? setup ?

Screening



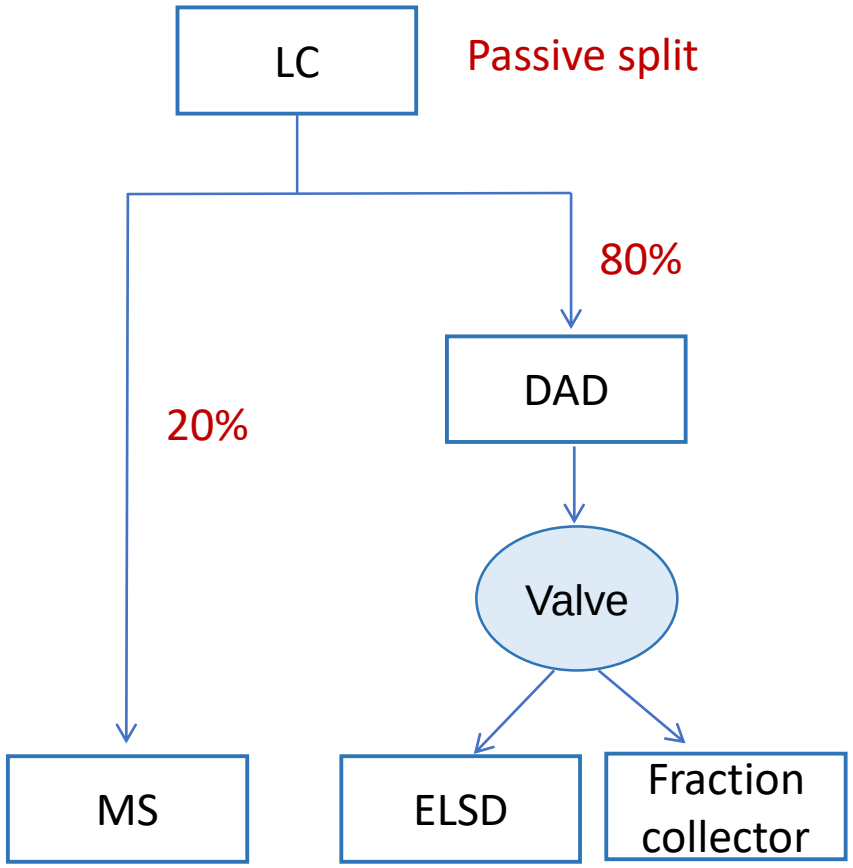
- ✗ MS signal ↓ , S/N ↑
- ✗ Leaking
- ✗ Solvent consumption



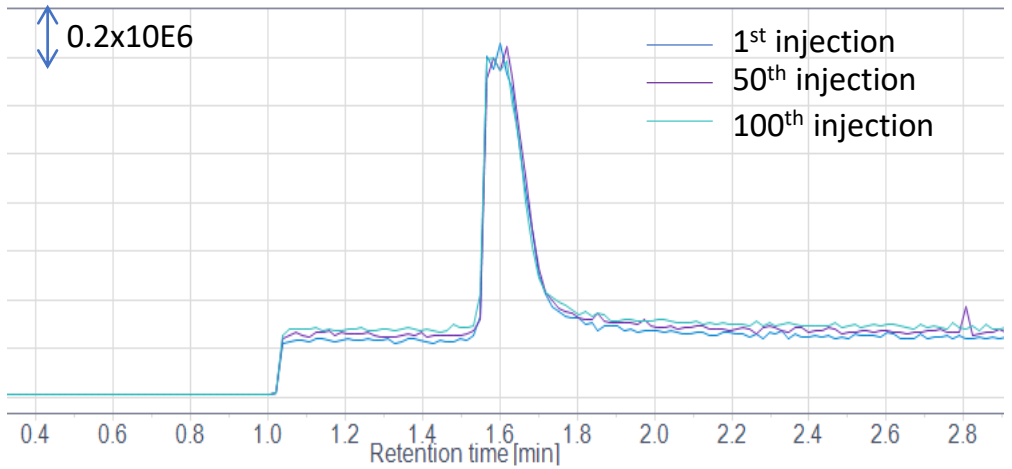
Mass spectrum of Flurbiprofen 10mM with flow modulator

Conditions:
A: H2O 0.05% acetic acid
B: ACN 0.05% acetic acid
Isocratic 70% B : 30% A
Flowrate: 1mL/min
$V_{injection}$: 1uL

Screening



- ✓ MS signal, S/N
- ✓ Leaking
- ✓ Solvent consumption



Conditions:
A: H2O 0.05% acetic acid
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Isocratic 70% B : 30% A
Flowrate: 1mL/min
$V_{injection}$: 1uL

Mass spectrum of Flurbiprofen 10mM obtained by passive split

Sample preparation

- One sample plate is transferred through all workflow
- Sample plate quality and security during transportation
- Adaption to all methods/ instruments (size, volume, concentration, solvent, etc.)
- Automation of many preparation steps
- Combination of all steps in one platform

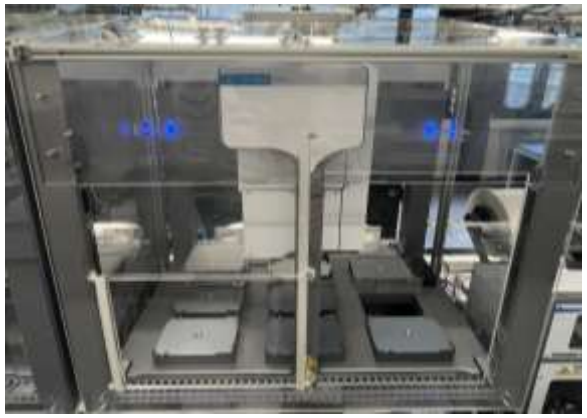
For example:

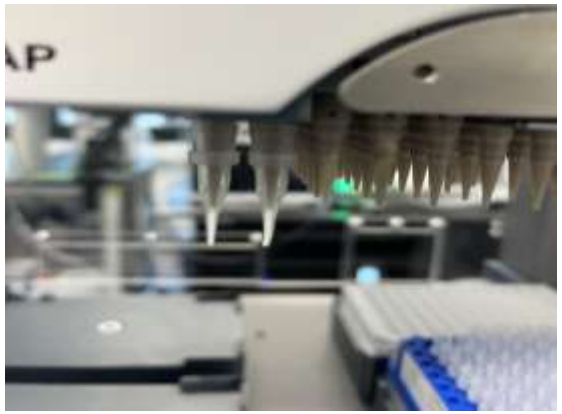
+ FTIR (10-20mM), UV (0.05mM)

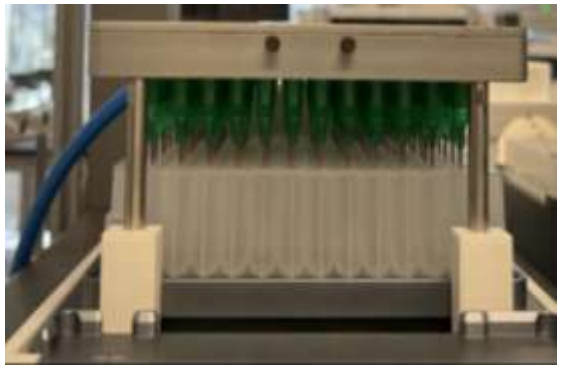
+ Deuterated solvent for NMR and normal solvent for FTIR and HR-UV

+ Low concentration sample collected from LC □ SFC

Sample preparation







-QR labelling

Centrifuge

Sealing

Stock

- 2 Agilent Bravo systems

- Building some accessories/materials if needed
- Hood installation for the volatile solvents

Characterization

- 1 semi prepLC

- 1 NMR 400 MHz

- 1 HR UV

1FTIR

- 1 SFC-IM-QTOF

Multidimension characterization

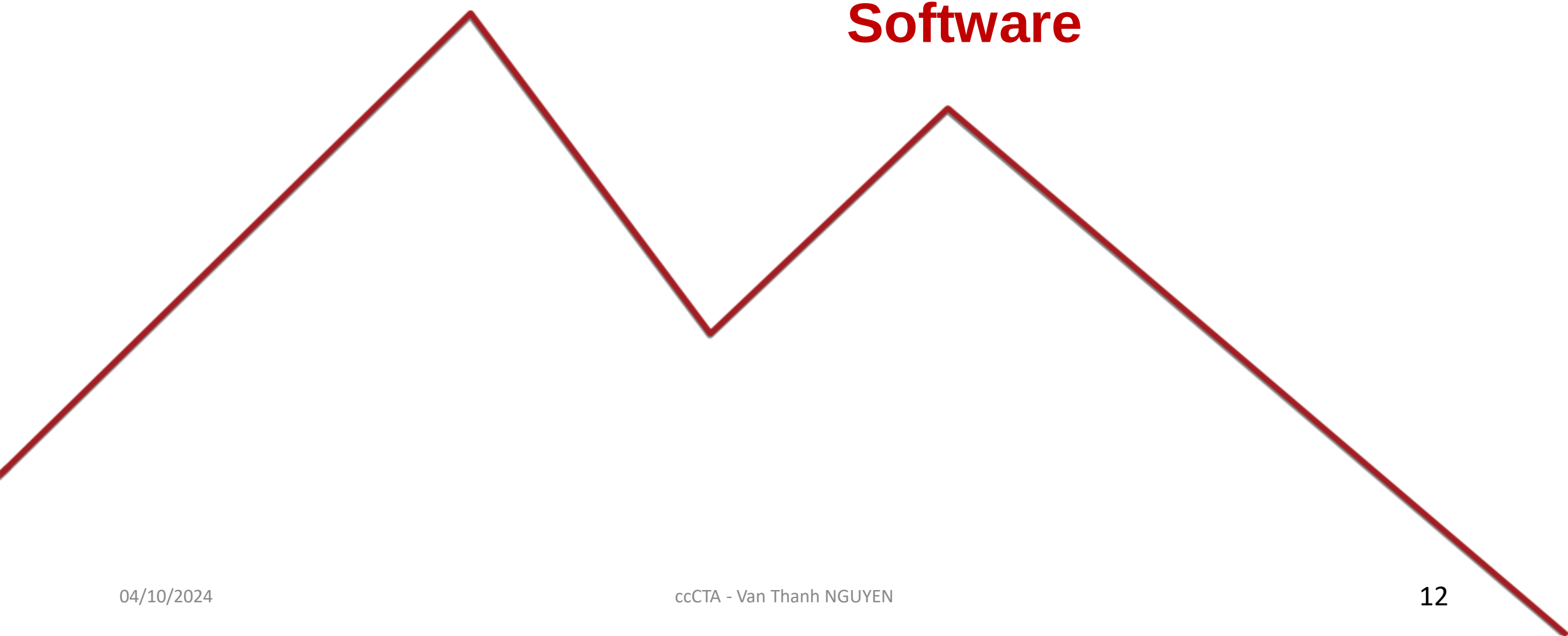
- Liquid flow transfer (flowrate, pression, etc.)
- Washing flow cells
- Different concentrations and solvent
- Automation of analytical instrument
- IM-QTOF automation (calibration, tuning, data processing)

Characterization

- OMNIFIRE

- Automated sample preparation for characterization
- Connected to the output of prepLC, collecting and splitting the outgoing flow into 96-well plates
- Transferring liquid samples to UV and IR flow cells and washing by nitrogen gaz
- Transferring liquid samples to an NMR sample tube and sealing.

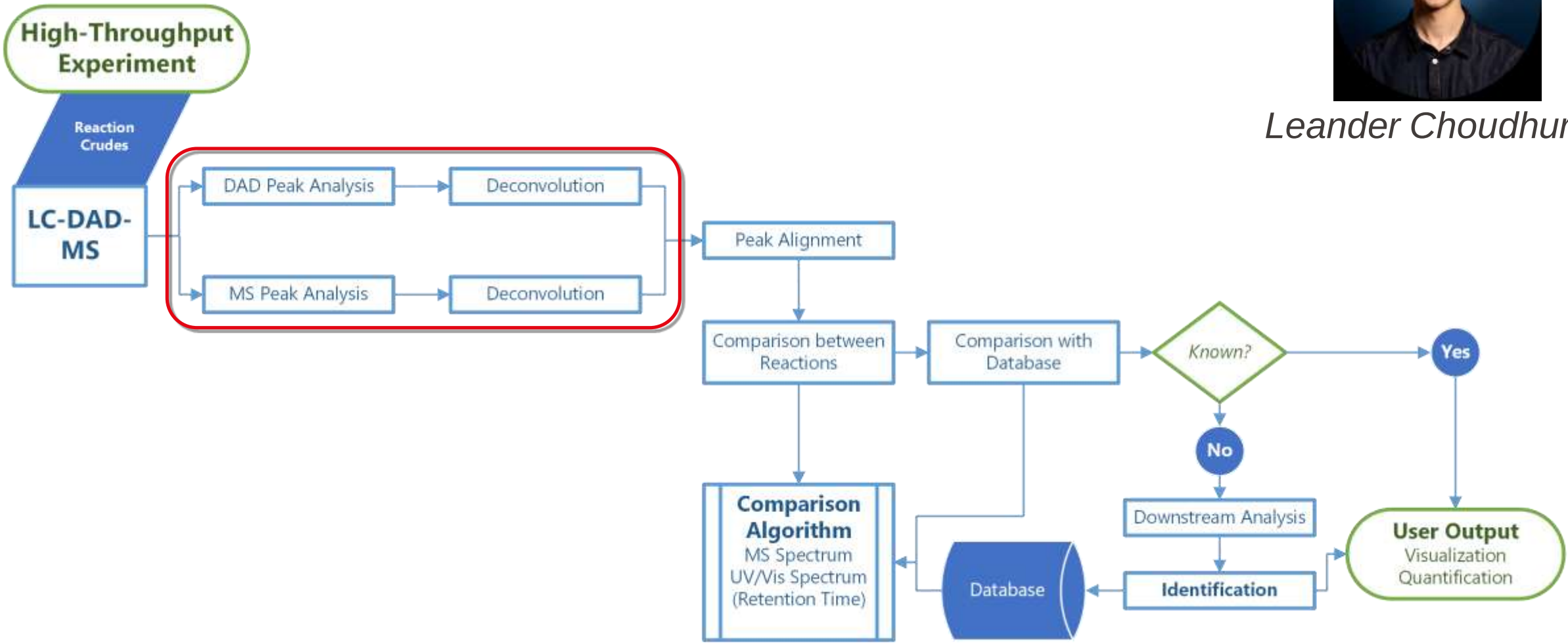
Software



- Same data format exported for all (CSV, ASM, JCAMP) not possible
- The complexity of management for the different formats data processing
- Complicated control for some instruments, unavailable or limited API
- Software are often proprietary
- Adaptation to each software

Sample Selection Algorithm

Leander Choudhury



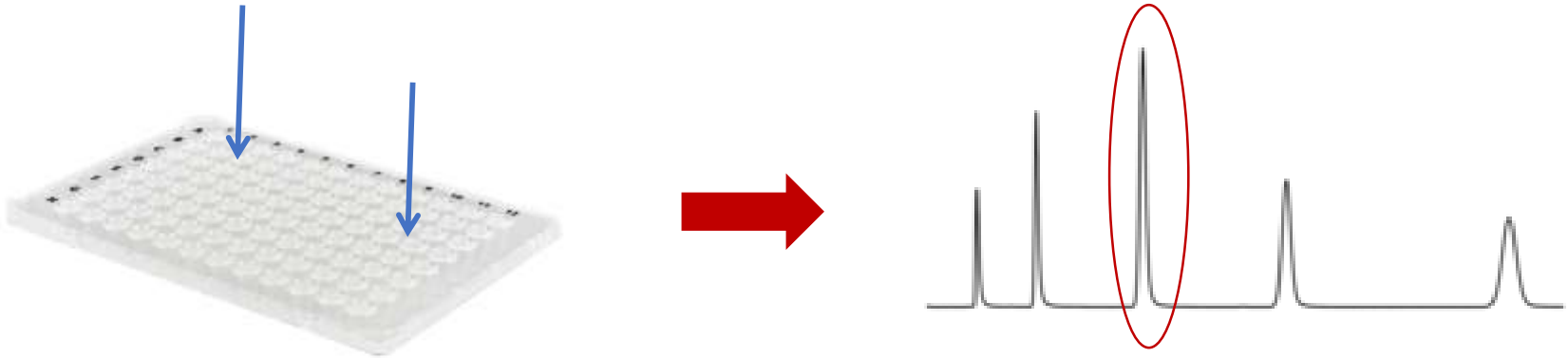
Challenges and perspectives

- Peak quality and data resolution

 Deconvolution

 False negative/false positive

- Include ELSD
- Communicating with lab scheduler & prep. LC



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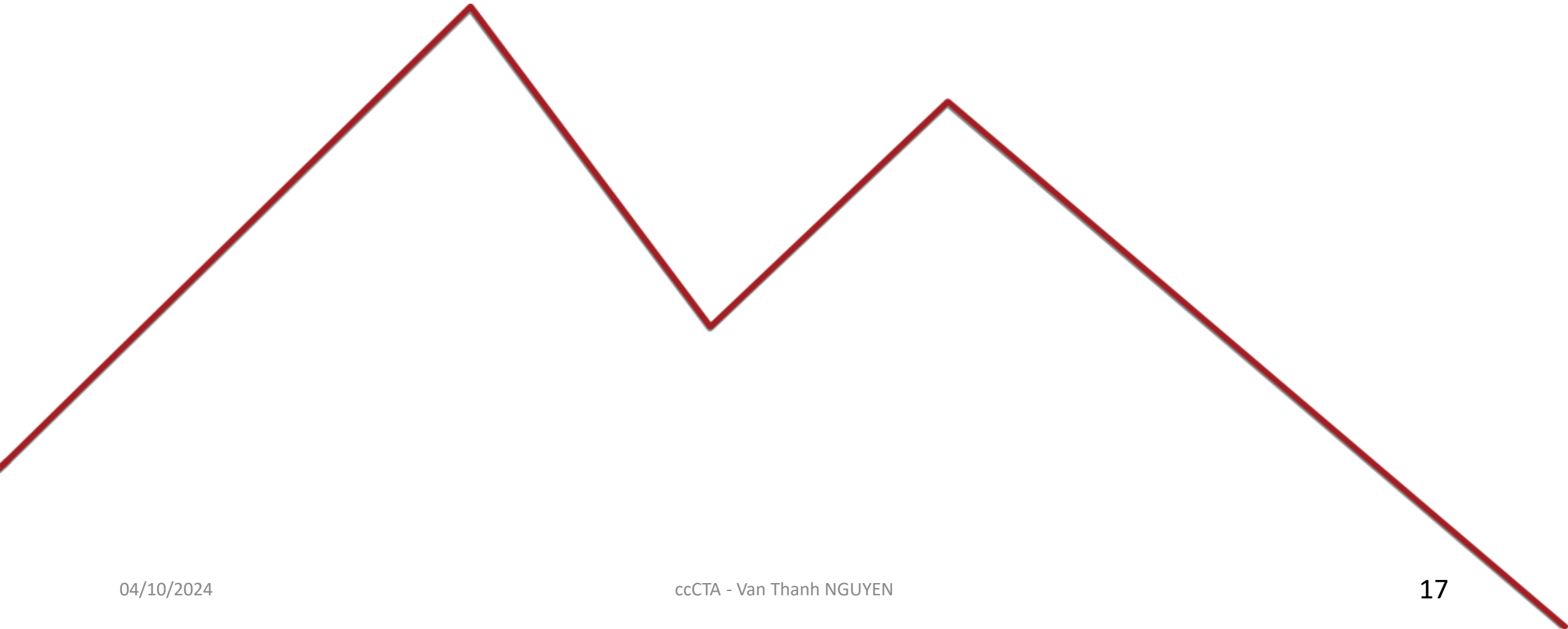
Sali Abdiraimi

Nathalie Bui

Enzo Venancio

- Trainees

Maël Löwensberg

Célia Moenho Dos Anjos

THANK YOU FOR LISTENING