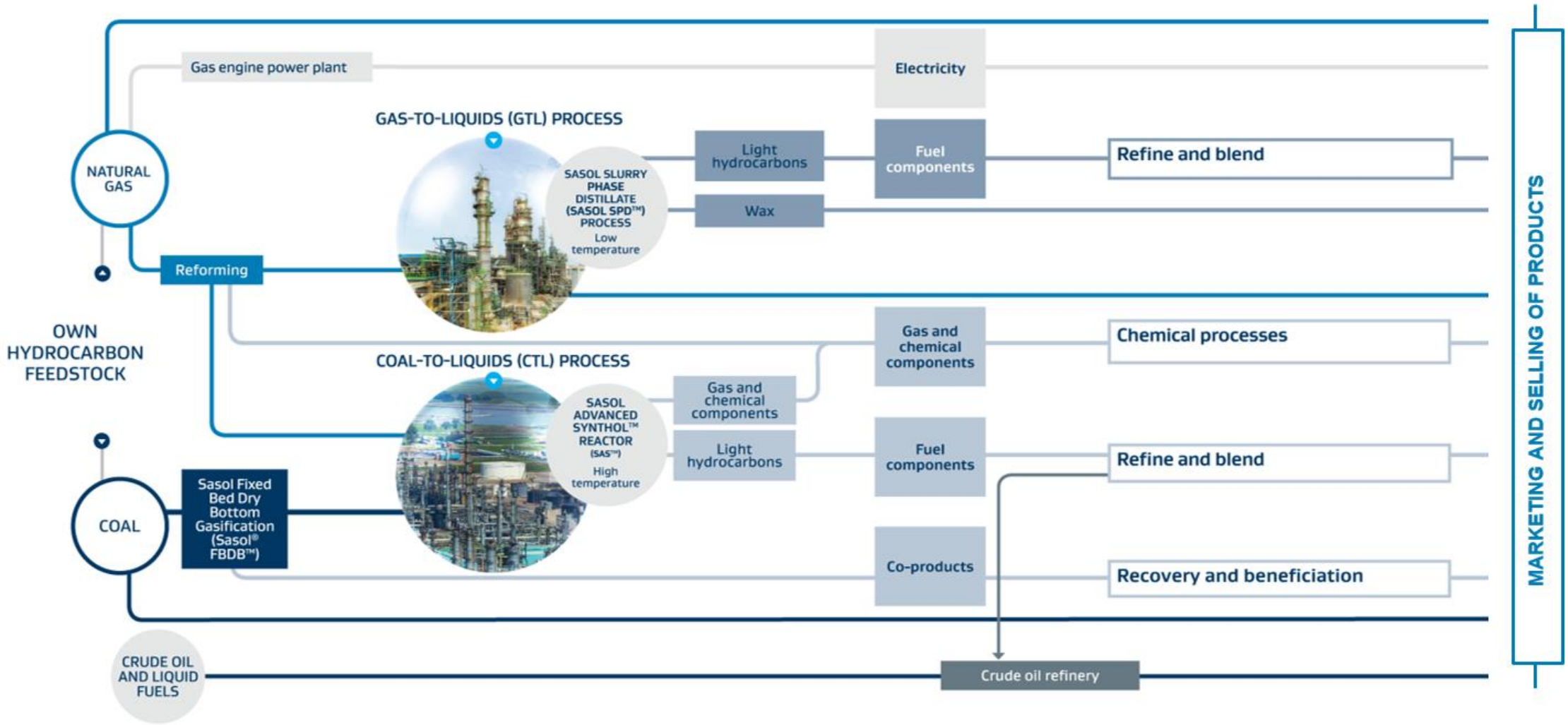
Analytical Chemistry ~ The unsung hero of Process Chemistry

Royal Society of Chemistry co-learning event ~ Process and analytical chemistry, there are more similarities than you think!

Dr AC Ferreira

7 February 2024

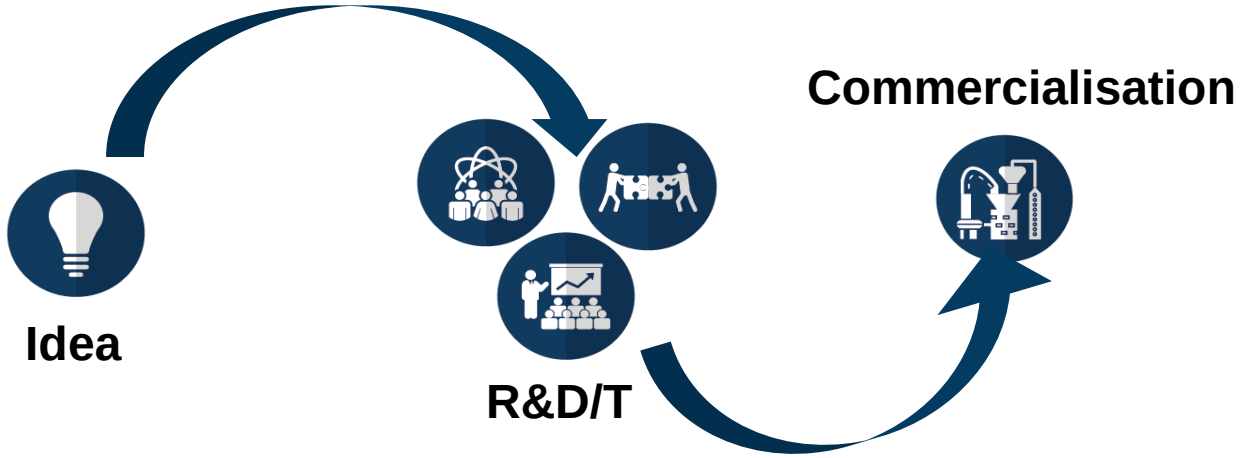
Sasol Process



Balancing People, Planet, Profit on our pathway to Net Zero

From Idea to commercialisation ~ multiple role players

Multi-disciplinary and diverse teams yields best results



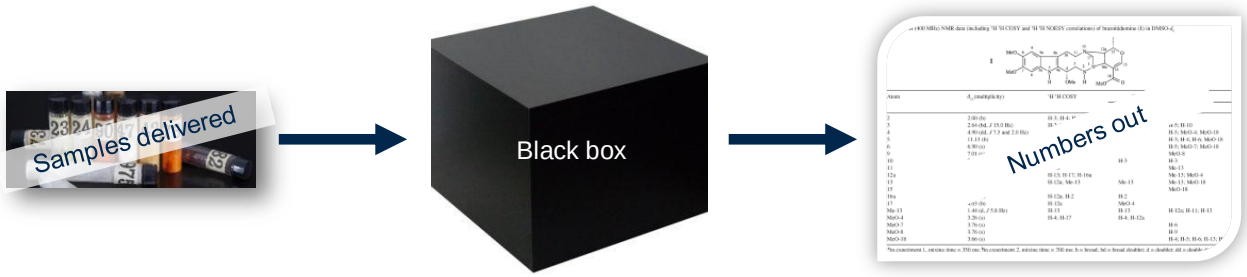
- Idea: FT Catalyst development – producing targeted hydrocarbon products e.g. wax, diesel, synthetic aviation fuel, etc.
  - Process Chemist: Develop and evaluate various catalysts to produce targeted products at maximum selectivity, yield and little to no by-products
  - Analytical requirements:
    - Gas composition/impurities, trace elements, poisons, etc.
    - Catalyst: Materials characterisation of physical and chemical properties, product analysis [critical for selectivity], etc.
    - Products: composition, impurities, etc.
    - Analytical manuals for routine laboratories

"When you can measure what you are speaking about, and express it in numbers, you know something about it, when you cannot express it in numbers, your knowledge is of a meager and unsatisfactory kind; it may be the beginning of knowledge, but you have scarily, in your thoughts advanced to the stage of science." **Lord Kelvin**

A bias view on analysis

Are the role players aligned w.r.t the end goal?

- Process Chemist Scientist:
  - Request: Provide composition information of process feed and product/ catalyst systems, etc.
  - Can I trust the analysis? I will first ask this question before I ask anything else?
  - Non-sensical analysis – why does the analytical scientist provide me with information I can't use?
  - OR I will take analysis as truth and design solution – large cost.



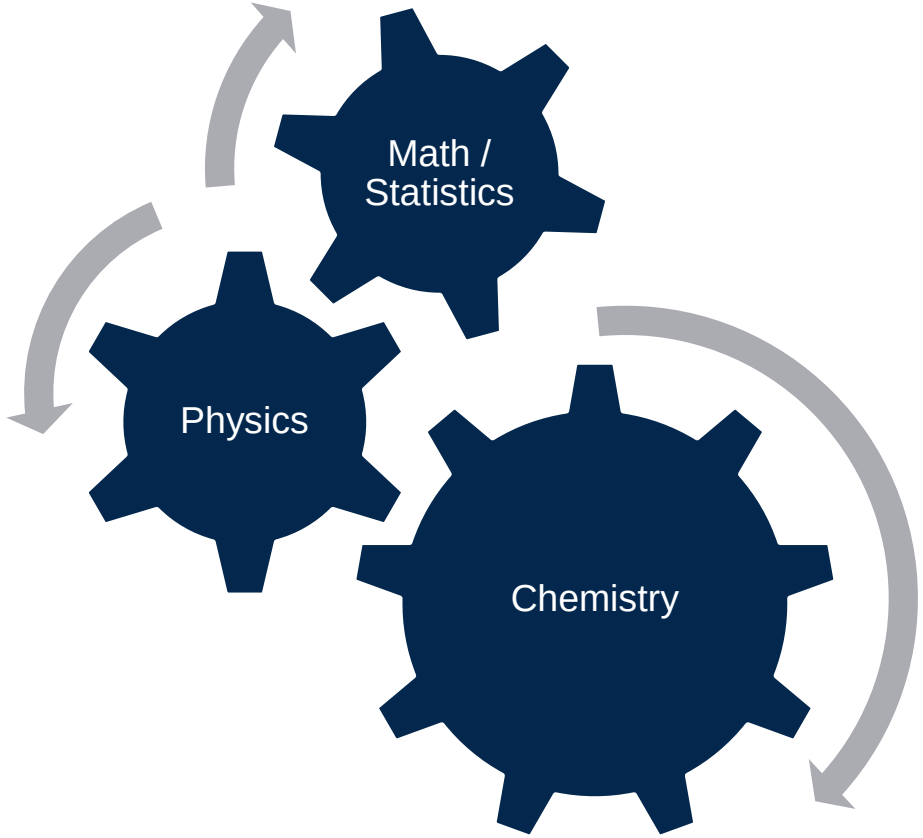
- Analytical Chemist/Scientist:
  - Will the analysis you requested answer your question?
  - Representative sample?
  - Sample matrix – critical for sample preparation
  - What does the number translate to
  - 80:20 principle "when is good enough, good enough?

Analysis of trace elements in Synthesis gas

How difficult can it be?

Example of analysis required [not all inclusive]:

- Solids in gas
- Nitrogen,
- Sulphur,
- Oxygen,
- Halides,
- Trace metals
- Etc, etc, etc……



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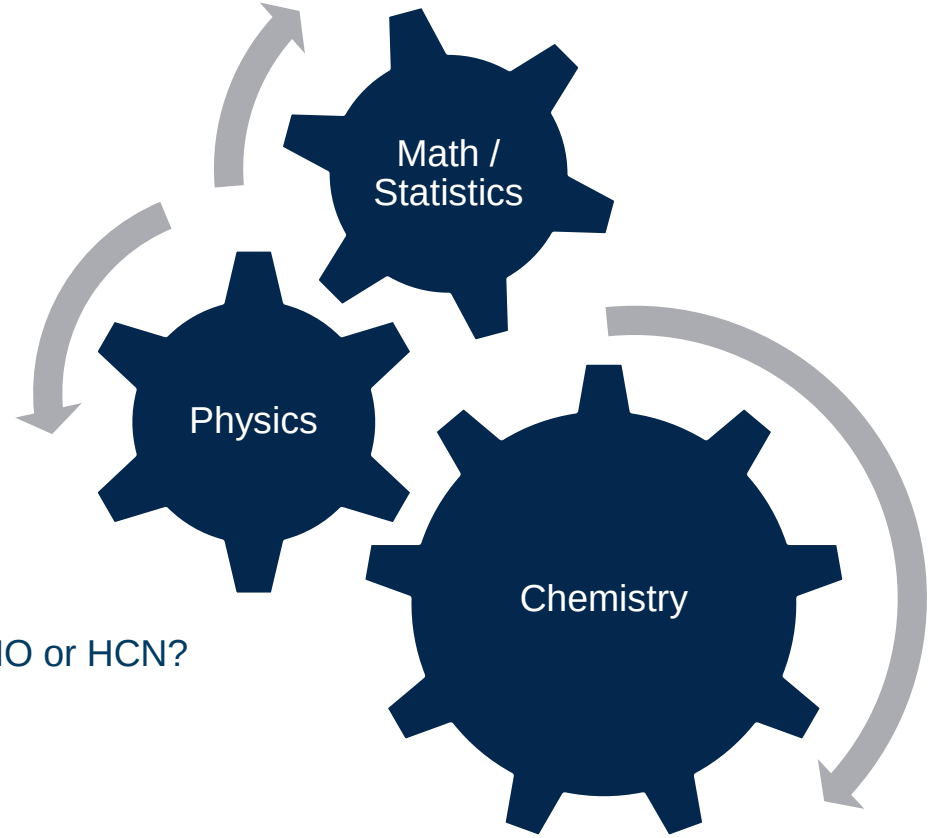
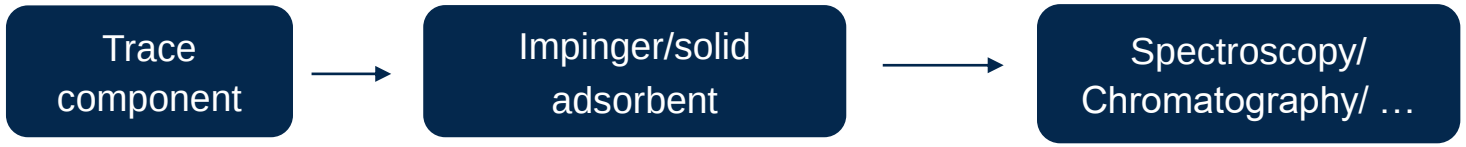


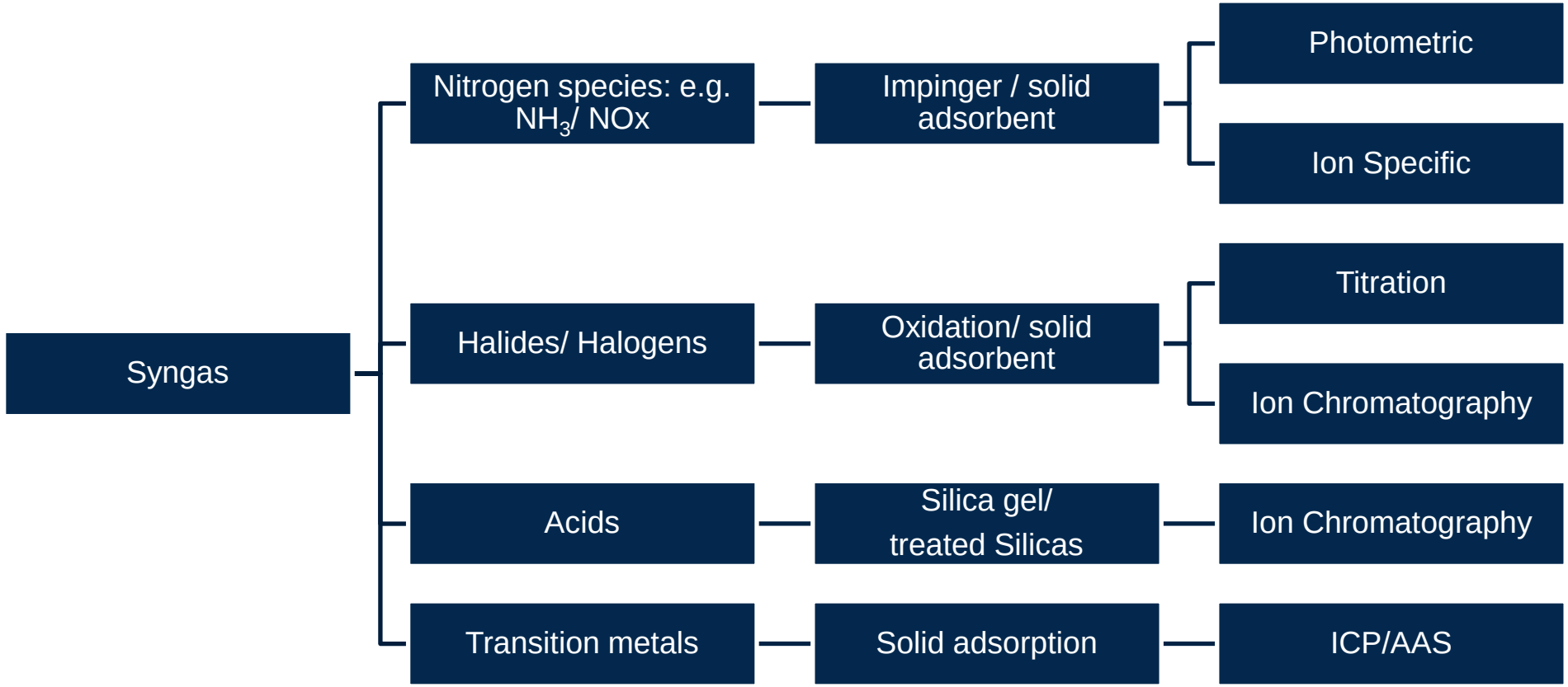
As Analytical Scientist:

- Questions, Questions, Questions
- Detection requirements, composition of trace elements e.g. NO or HCN?
- Proof of concept – study the literature!
- Implementation plan [Potential suppliers, sampling, etc.]
- Testing
- QC/ Reporting



Synthesis gas analysis (cont…)

General methodology



Analysis of multiple components within a gas stream requires one to go back to fundamentals of Science

Analysis of trace elements in Synthesis gas (cont…)

Nitrogen dioxide analysis ~ potential solution

- Literature examples: *A Micro-impinger Sampling Device for Determination of Atmospheric Nitrogen Dioxide*
  - µ-impinger ~ NO_2 concentrations < ~8 ppb
  - NO_2 + absorbing reagent = red-violet color
  - absorbance measured at 540 nm (UV-1800, Shimadzu, Japan),
  - Reference: unexposed reagent

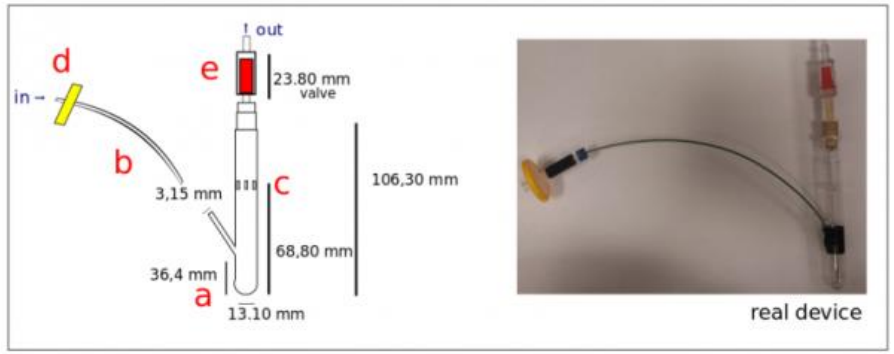


Diagram of µ-impinger bubbler (a) µ-impinger bubbler tube, (b) PEEK tube, (c) glass finger, (d) Teflon filter and (e) check valve

Passaretti Filho *et al.,* Aerosol and Air Quality Research, 19: 2597–2603, 2019

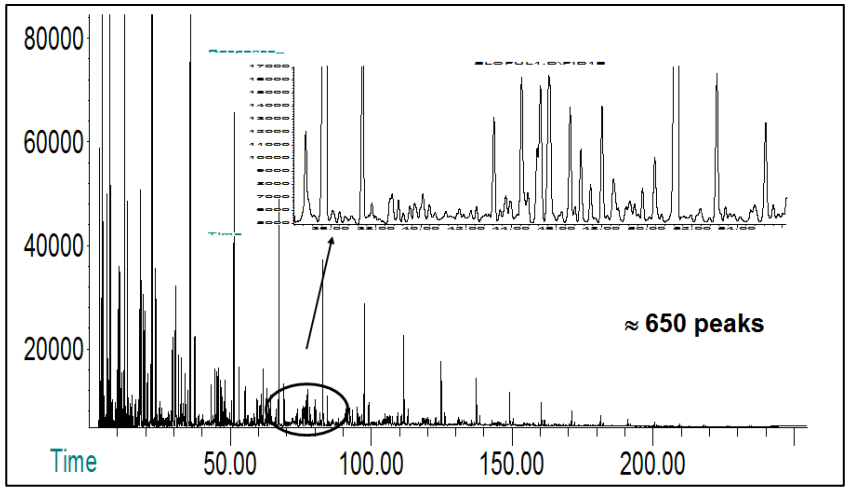
Question: How will you apply this to synthesis gas? What are the limitations?

Example: predict and optimize linear paraffins in hydrogenation process

FT process → Light condensate fraction → Hydrogenation → Products e.g. Petrol, Light diesel, etc.

Complex product slate

Important specification = Mass % linear paraffins



GC-FID of light condensate fraction

Data from 1 D GC is not fit-for-purpose

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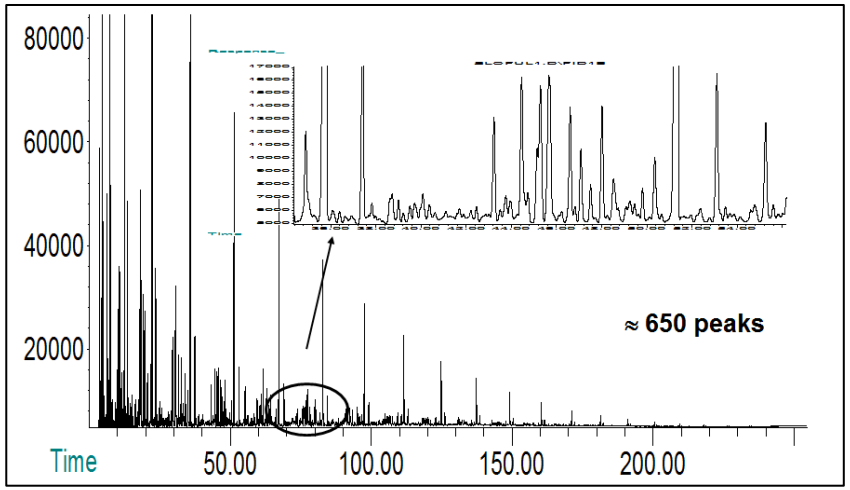
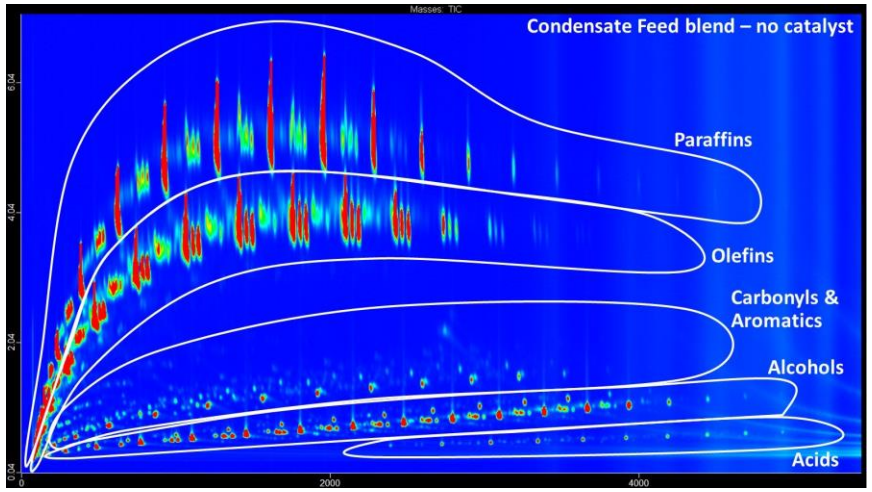


GC-FID of light condensate fraction



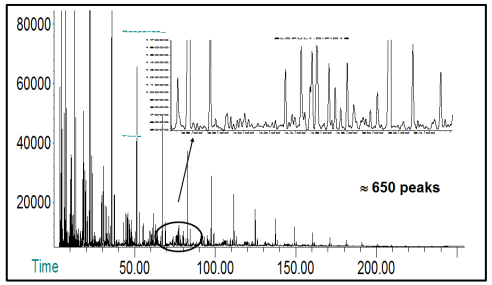
GCxGC of light condensate fraction

GC x GC analysis ~ also not fit-for-purpose

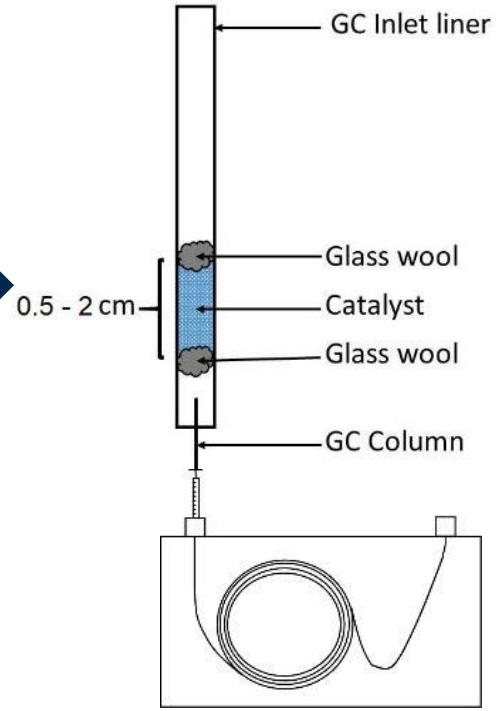
Example: predict and optimize linear paraffins in hydrogenation process (cont…)

Solution: Inlet hydrogenation GC-FID Analysis

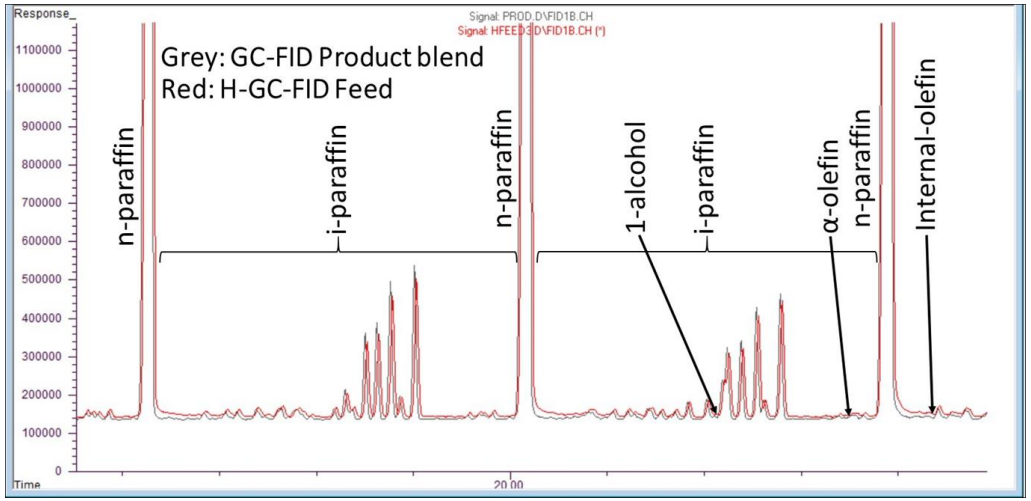
Feed



Hydrogenation +
1D GC Analysis



% linear paraffins in feed



H. Potgieter, R. Walmsley, "*Inlet hydrogenation gas chromatography to predict mass % linear paraffin content*", *Journal of Chromatography A, 1680, 2022, 463441*

Elegant analytical solution to quantify % linear paraffins in hydrogenation feed

Key learnings as a Process Scientist converted to Analytical Scientist

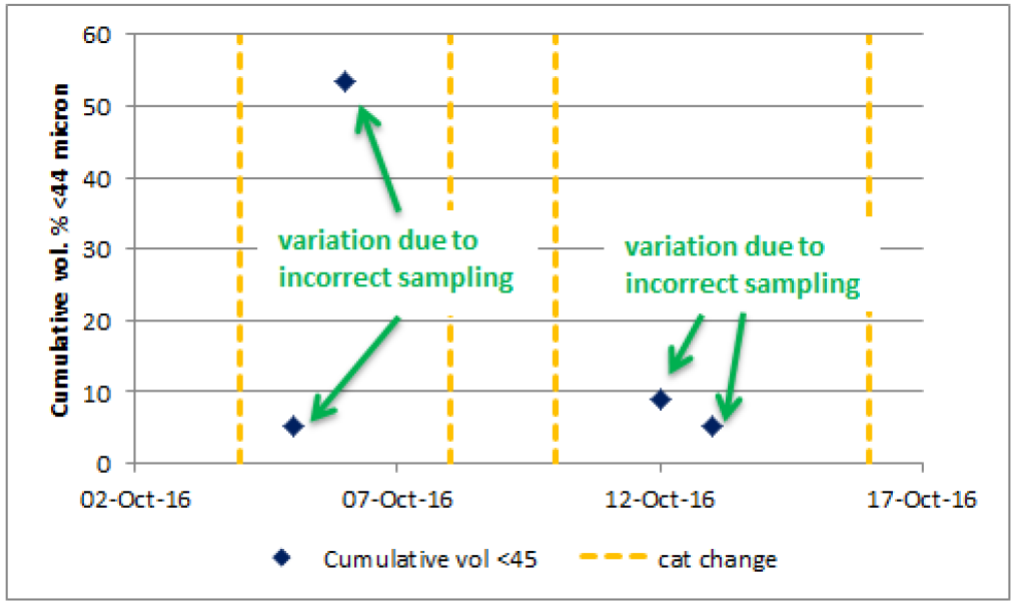
- Communication, communication, communication….
- Listen with the intend to understand first – then question
- Information is key – share as much about your process as possible
- Your solution is not necessarily the best
- Understand what the numbers mean vs your requirements
- It all starts with correct sampling
- Analysis paralysis is real….
- Quality Control saves time, money and resources if implemented correctly

What is your problem statement/ requirement/ research question?

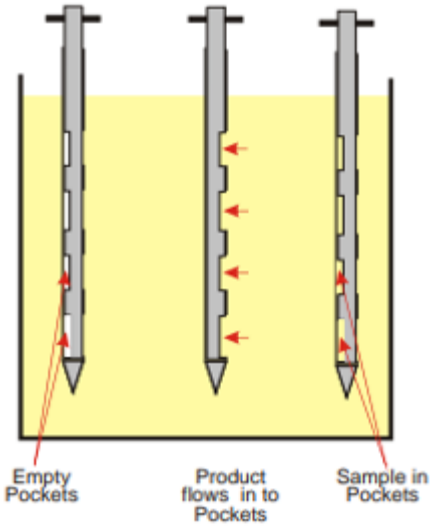
Analysis paralysis….

How to prevent this syndrome from an Analytical perspective

- **Sample integrity/ representative sampling using a pocket sampler**



Particle Size distribution



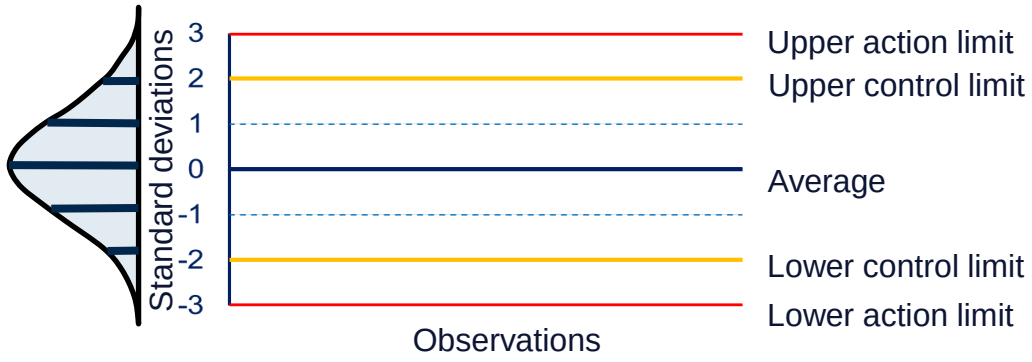
Example of a Pocket sampler

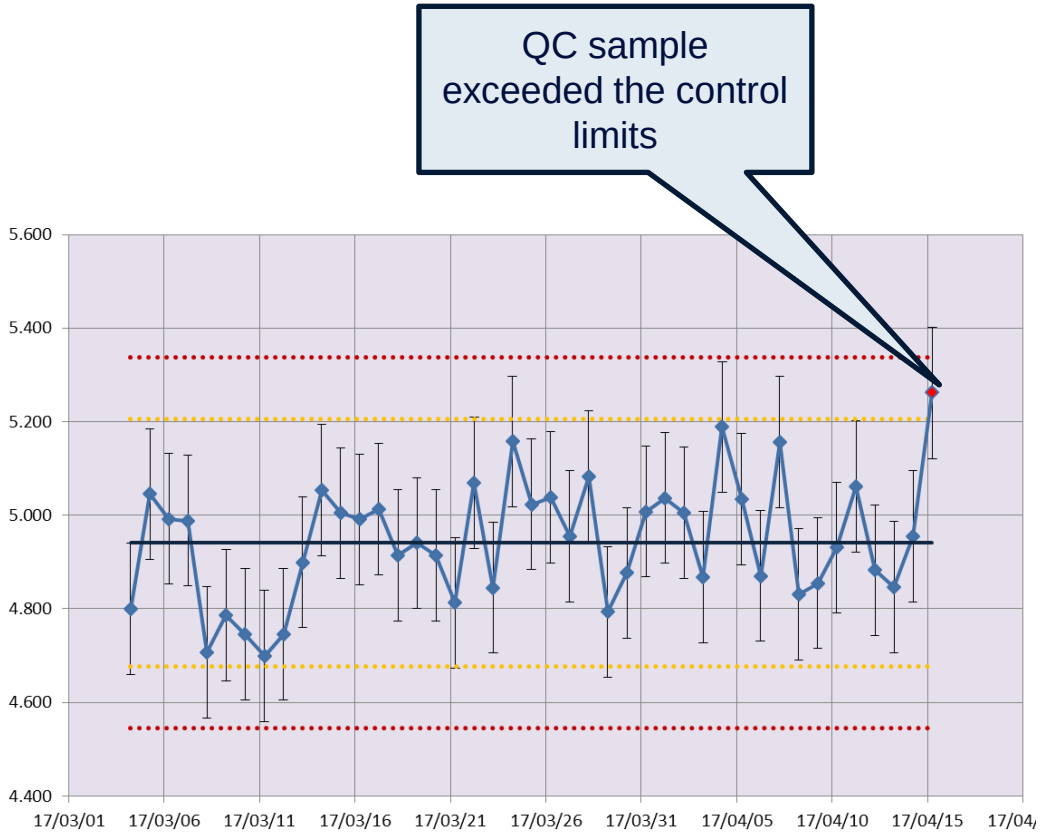
Sampling requires a scientific and systematic approach

Analysis paralysis….

How to prevent this syndrome from an Analytical perspective

- **Validate** and **verify Analysis:**
  - Validate to show that the method is fit for its purpose.
  - Verification is a continuous process
- **Control Charts:**
  - Determined by method validation
  - Decision limits are based on analytical requirement specification





Validation/Verification and QC enables quick and informed decision making in operations

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SASOL