From characterisation to validation, a journey through MSc applied analytical chemistry.

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Department of Chemistry

- Careers

  Agriculture and Food.

  Environment.

  Toxicology.

  Oil and Gas.

  Pharmaceutical.

  Personal care.

  Forensics.

- Aim

“This MSc seeks to train the next generation of analytical scientists in state-of-the-art methods”

“is designed to provide comprehensive training in analytical chemistry and its implementation. A thorough understanding of ***error analysis***, ***data processing*** and ***data presentation*** is at the foundation of this programme”.

- Exercise.

From the following statements decide which is *qualitative*, *quantitative*, *characterisation* or *fundamental* analysis.

A. A waste incinerator is suspected of releasing more than the legal limit of dioxins into the atmosphere.

B. An art gallery suspects a masterpiece was switched to a forgery.

C. Airport security needs a more reliable method for detecting explosives in luggage.

D. The loss of metabolites of a new drug through excretion in urine need to be determined.

- Importance of measurement.

Identification of impurities / contaminants.

- **regulatory limits.** ✓ ✗ ?
- **batch quality.**

Characterisation of a new substance.

- **chemotherapy.**

General assay – content or purity.

- **process improvement.**
- **long term trends.**

- Method performance characteristics.

Limit of detection LOD (MLD) - *sensitivity*

Limit of detection LOQ (MLQ)

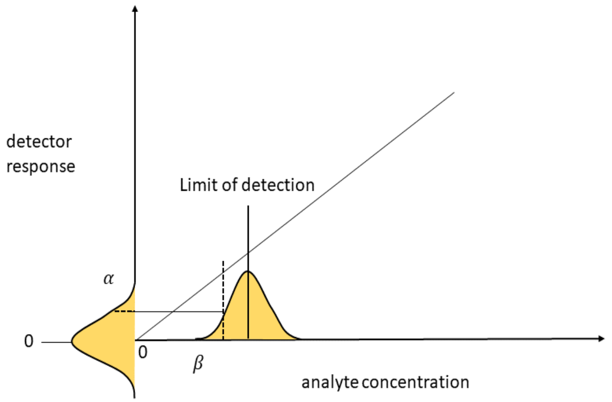
Bias - *accuracy*.

Precision (homogenous samples)

 Repeatabilty

 Reproducability

Ruggedness – *method control*



- Chemometrics

Chemistry, chemical engineering, biochemistry, medicine.

Factors, responses and variables.

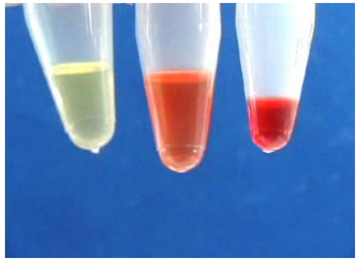
It was noted that an increase in foaming was accompanied by an increase in alcohol content.

- Chemometrics

Statistical experiment design (Design of Experiment)[1].

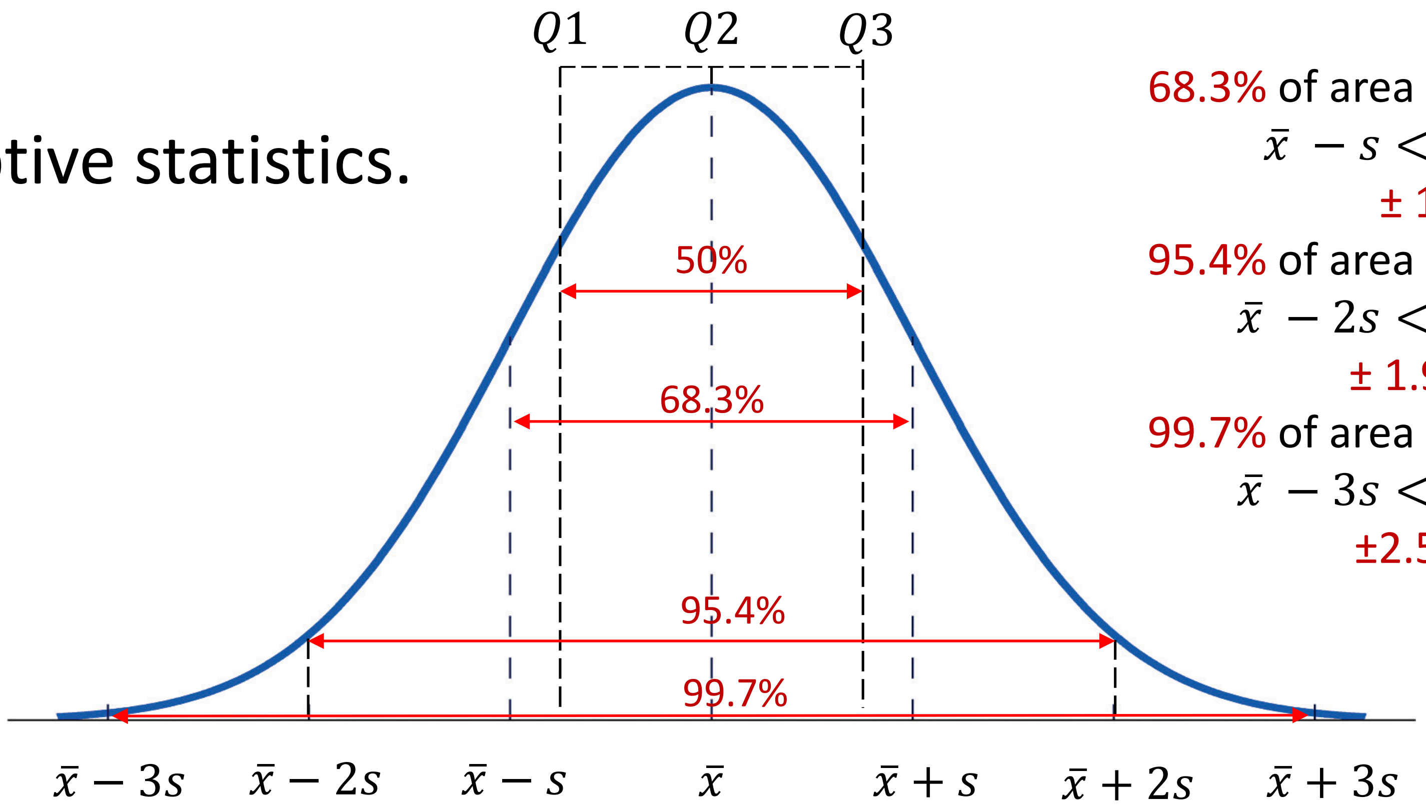
Sampling	• Representative. • Variance.
Experiment design	• Extraction, clean up, pre concentration. • Data preprocessing.
Factor analysis	• Variable reduction, pH, temperature… • Controlled, uncontrolled, masquerading.

- SAMPLING



• Data Analysis and Theory.

Descriptive statistics.



68.3% of area under the curve;

$\bar{x} - s < \bar{x} < \bar{x} + s$

$\pm$ 1 sem

95.4% of area under the curve;

$\bar{x} - 2s < \bar{x} < \bar{x} + 2s$

$\pm$ 1.96 sem

99.7% of area under the curve;

$\bar{x} - 3s < \bar{x} < \bar{x} + 3s$

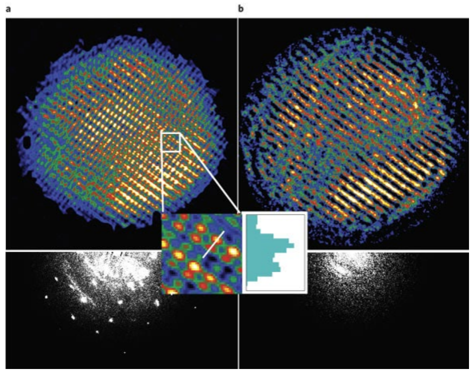
$\pm$2.58 sem

- Data Analysis and Theory: DESCRIPTIVE STATISTICS.

- Synthesis of cadmium sulphide crystal quantum dots by bubbling hydrogen sulphide (H_2S) gas over a foam with cadmium ions.

- Diffractive imaging showed CdS quantum dot sizes ranging from 7 – 9 nm.

- Estimate $df\ (N-1)$, $\bar{x}$, s^2, s, cv and $s_{\bar{x}}$.



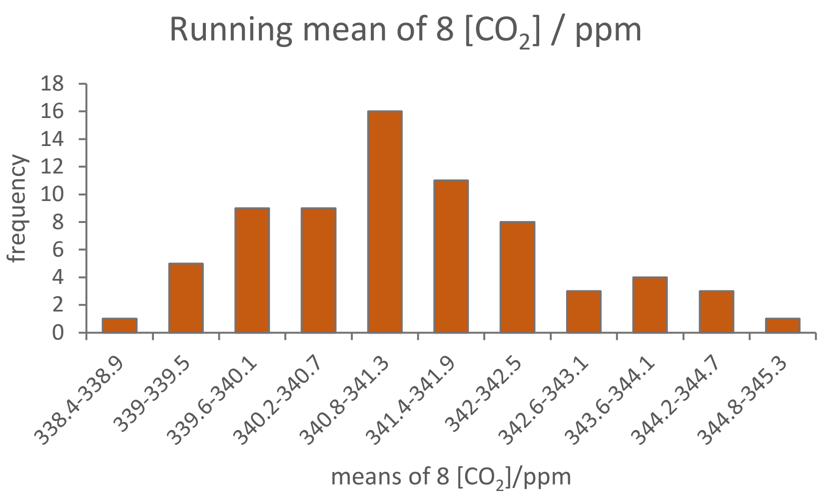
Huang W.J, Zuo J.M…*Nat. Phys. Letters.*, 2009, **5**, 129

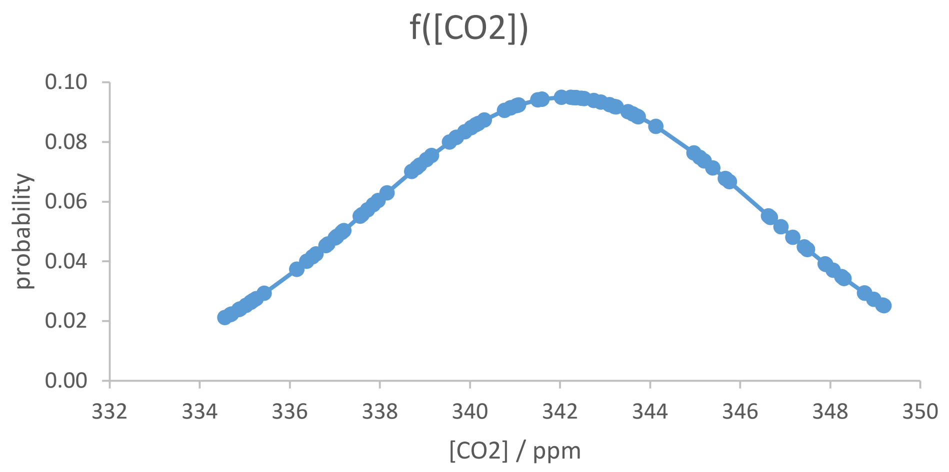
7.5 nm	9 nm	8.5 nm	8 nm	7 nm
7 nm	9 nm	7.5 nm	8 nm	8.5 nm
$\bar{x}$ =	s^2 =	s =	cv =	$s_{\bar{x}}$ =

- SIZE OF DATA SETS.....and DISTRIBUTION.

Atmospheric CO_2 at Mauna Loa in June – August in 1985: 334.6 to 348.7 ppm. Assuming a uniform distribution plot a;

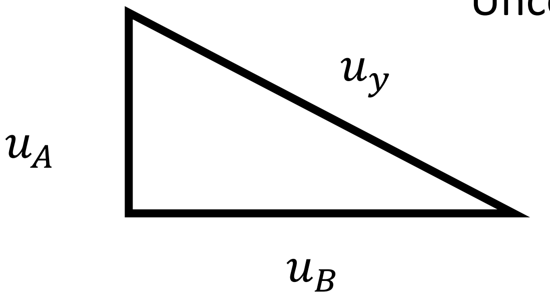
- histogram of the running means of eight observations.
- continuous normal distribution using the parameters.





• Error and Uncertainty

- Uncertainty estimate.



Uncertainty in response y;

$$u_y = \sqrt{u_A^2 + u_B^2}$$

$$\delta z = z\sqrt{\left(n\frac{\delta a}{a}\right)^2 + \left(m\frac{\delta b}{b}\right)^2 + \left(p\frac{\delta c}{c}\right)^2 + \left(q\frac{\delta d}{d}\right)^2}$$

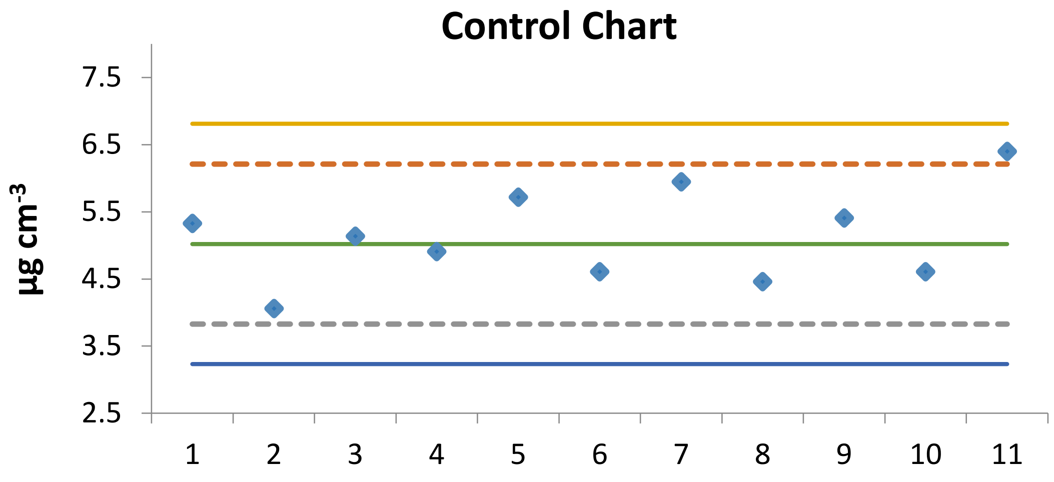
- Uncertainty budget.

	Contribution to uncertainty.
a	Intercept of calibration curve.
b	Slope of calibration curve.
c	Intermediate precision.
d	Concentration of calibration solutions.

Quality

ASSESSMENT – How? Planned set of activities, documentation. **ISO / IEC.**

CONTROL – What? Programme of activities, what is done. **Control Charts.**



- Inferential Statistics.

F variance ratio, **Z tests**.

Student t tests.

Levels of geraniol in a suspected fake perfume sample.

ANOVA.

Ochratoxin A levels in samples from bulk wheat storage.

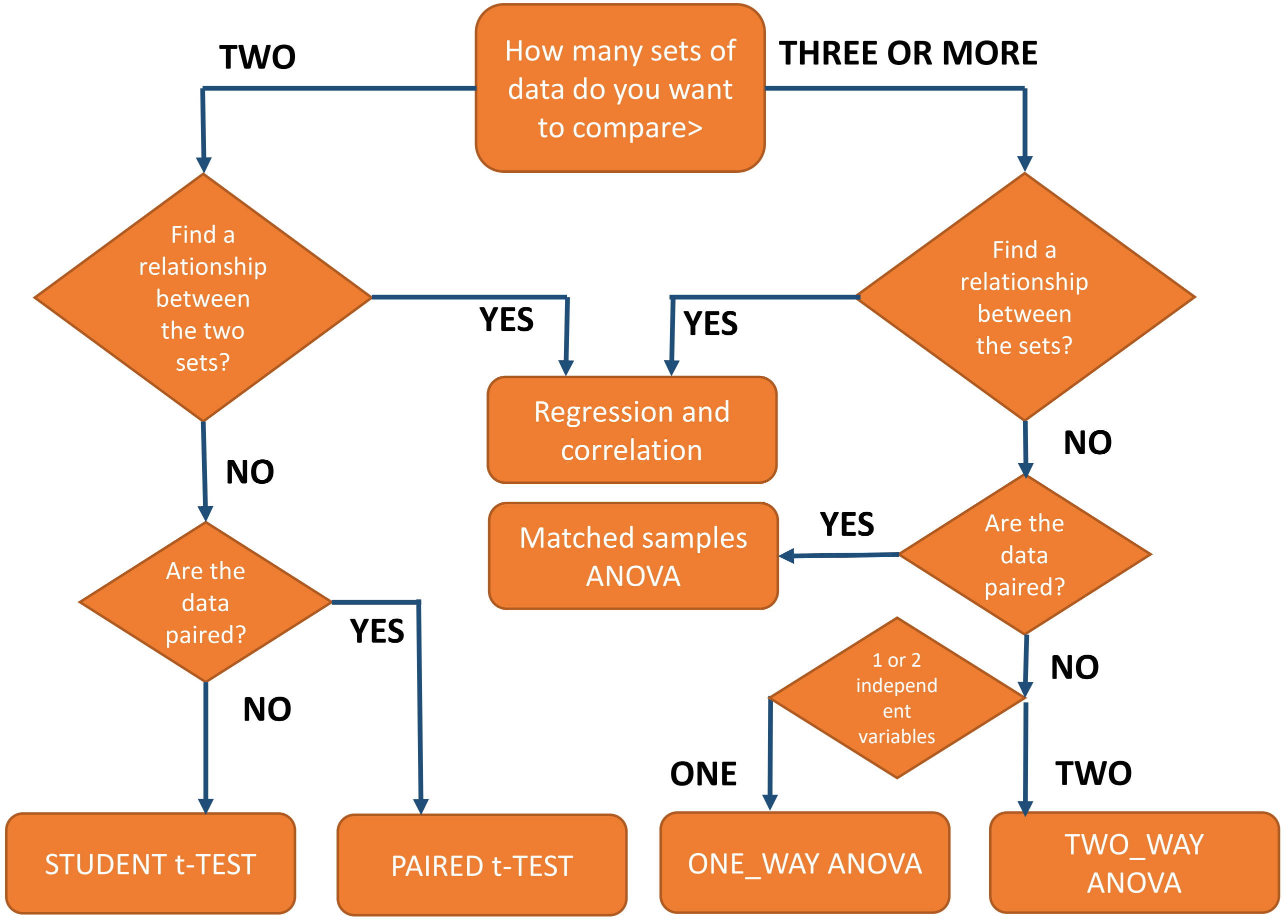
GLM – ANOVA.

Significance of difference between levels of acetaminophen glucuronide in different sections of a river.

Chi Square (χ^2).

Presence of uni and divalent cations in fermentation tank effect alcohol level.

* Parametric tests



- Student *t* test: Comparing two methods.

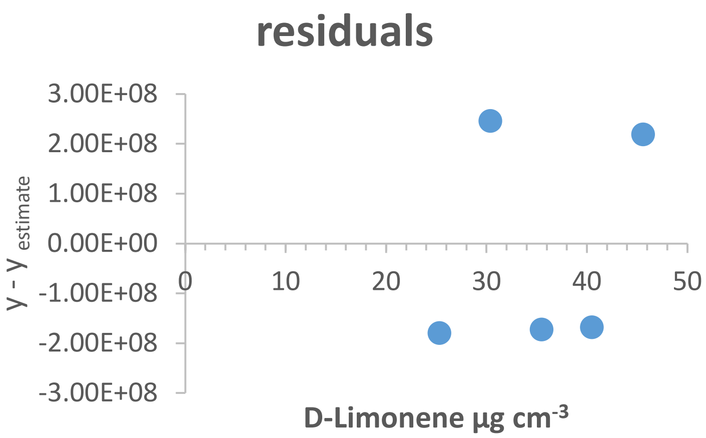
Vitamin B9 recovered from green kiwi fruit using 2 extraction methods.

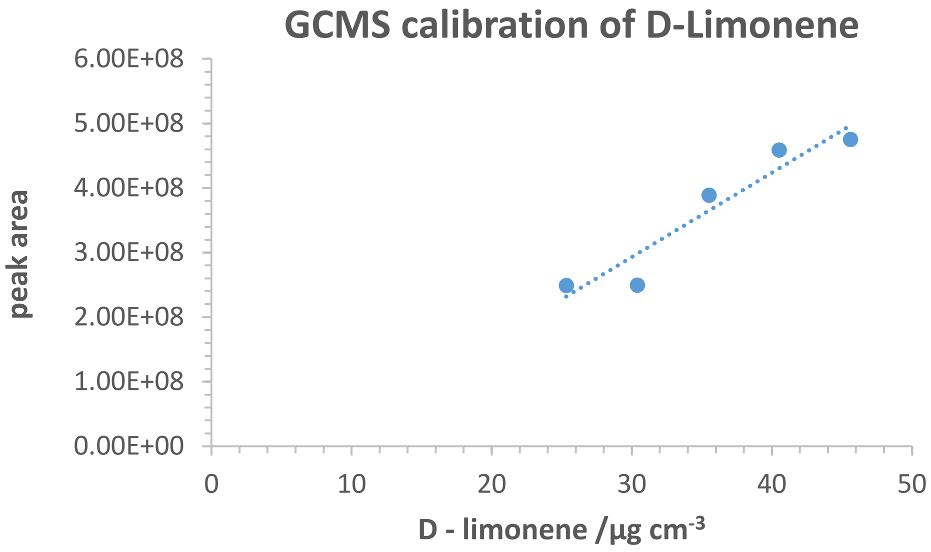
$$F \text{ test} = \frac{s_1^2}{s_2^2}$$

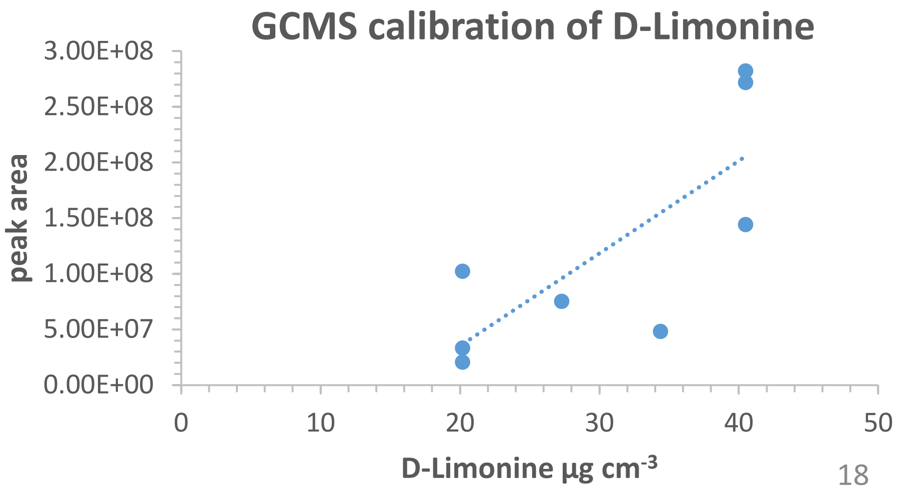
combined standard deviation, $s_c = \sqrt{\frac{s_1^2(n_1-1)+s_2^2(n_2-1)}{(n_1+n_2-2)}}$

$$t = \frac{\bar{x}_1-\bar{x}_2}{s_c\sqrt{\frac{1}{n_1}+\frac{1}{n_2}}} \qquad df = n_1 + n_2 - 2 \qquad t_{critical} < t_{estimate}$$

- Practical: Quantification of D-Limonene in perfume using GCMS.







- Data: Estimate and Discuss.

Uncertainty from:

slope of calibration, repeatability,

concentration of calibration solutions.

F test:

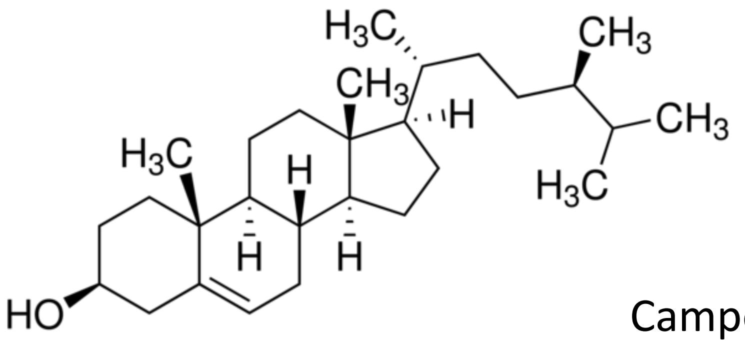
- variance ratio repeats of sample perfume versus real perfume.
- student data comparison in pairs.

t test:

two sample, two tail.

THEIR Method Limit of Quantification (MLQ), Bias.

- Steroids in Soya Milk – LCMS[3]



Campesterol

Extraction
- Liquid – liquid to
- liquid - solid.

Derivatisation
- Cholesterol oxidase.
- Girard P.
- Overnight.

Extraction
- SPE
- Washed and eluted x 3.

Analysis
- Thermo Accela Ion Trap LCMS[3]
- Hypersil Gold C18

- Arduino – Analytical Device Construction.

Sample

The analyte in a matrix: a mixture of potential interferants and solvent.

→

Transducer

Converts energy from one form to another. Chemical, electromagnetic.

→

Output

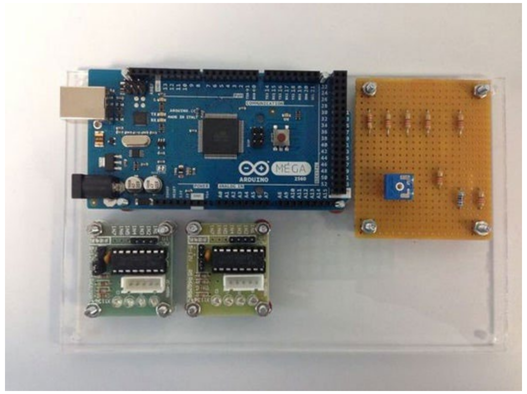
Display of data- raw and processed for interpretation.

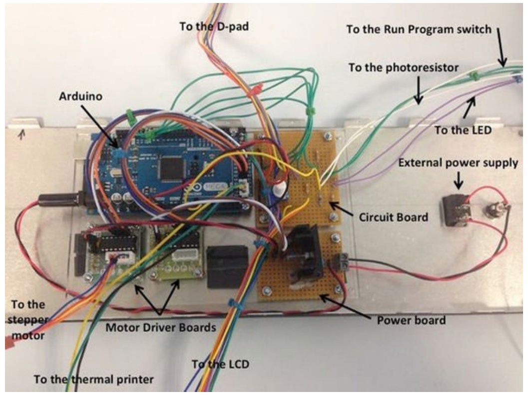
- Analytical Strategies - ARDUINO projects https://www.arduino.cc/.

Programmable boards, ***electronics – sensors***, mechanical parts and open source software (coding).

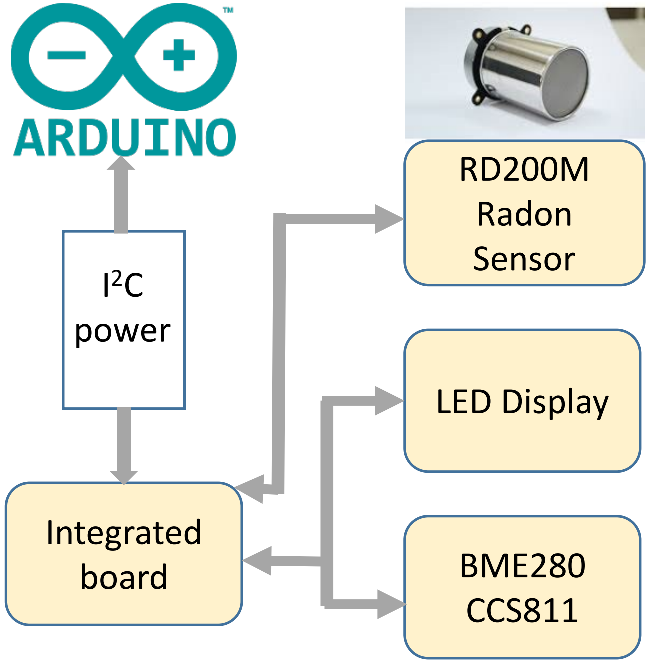
16 Steps to a spectrophotometer

https://www.instructables.com/id/An-Arduino-powered-microplate-spectrophotometer/

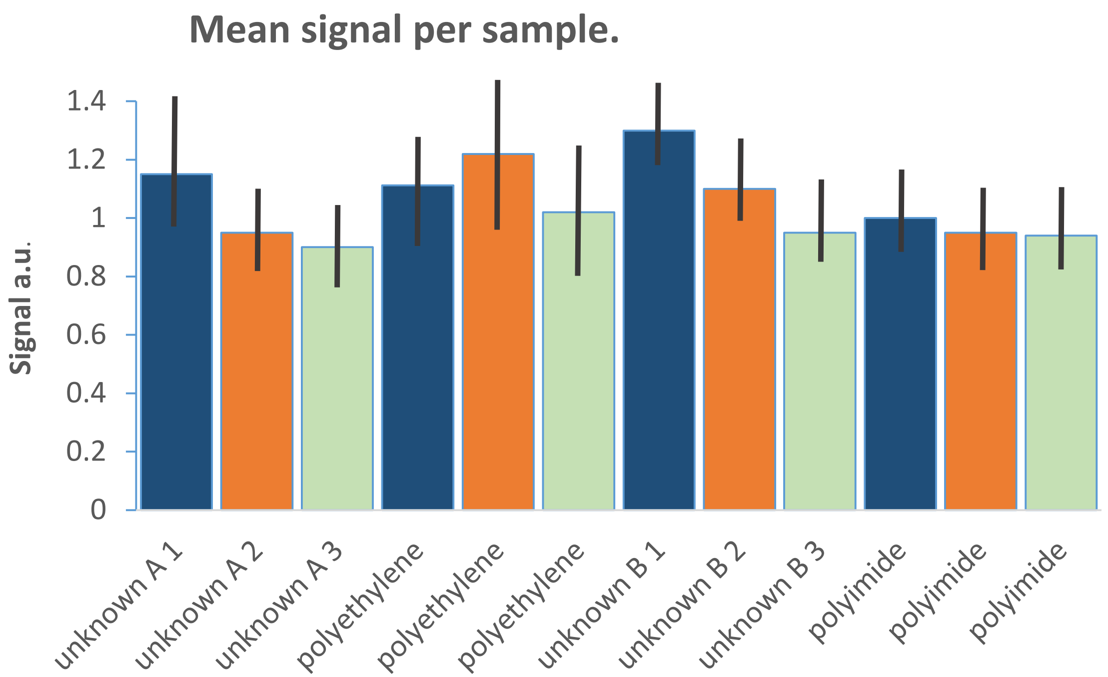




- Arduino - Apples to Radon.



- Arduino- NIR Plastics Identification.



Single Factor ANOVA.
$(\alpha = 0.05)$

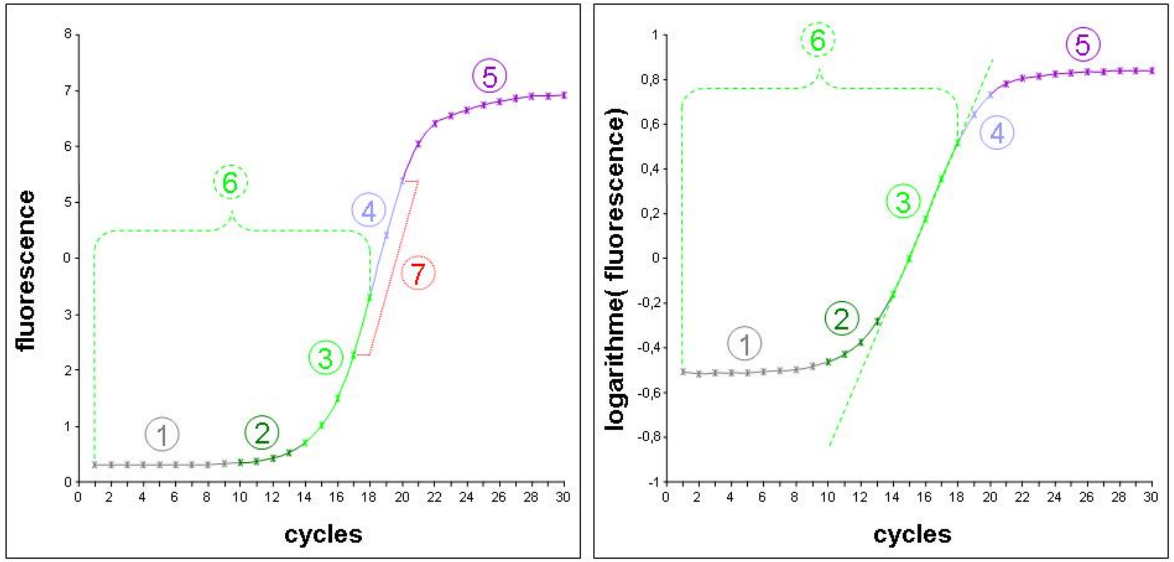
F	*p*	*F critical*
10.18	0.0016	3.68

?

• Analytical Strategies II:

Approach to Laboratory Diagnosis of Infection.

Source of diseases.
Metrology in microbial diagnosis.
Antimicrobial resistance.



By --Ygonaar 23:09, 7 March 2006 (UTC) - It's a graph create by Ygonaar with Power point, CC BY-SA 3.0, https://commons.wikimedia.org/w/index.php?curid=607322)

- Analytical Strategies II: Forensic Toxicology.

Drink and drug driving (abuse).
Action in the body.
Opioids
Analysis – GCMS
Case studies.





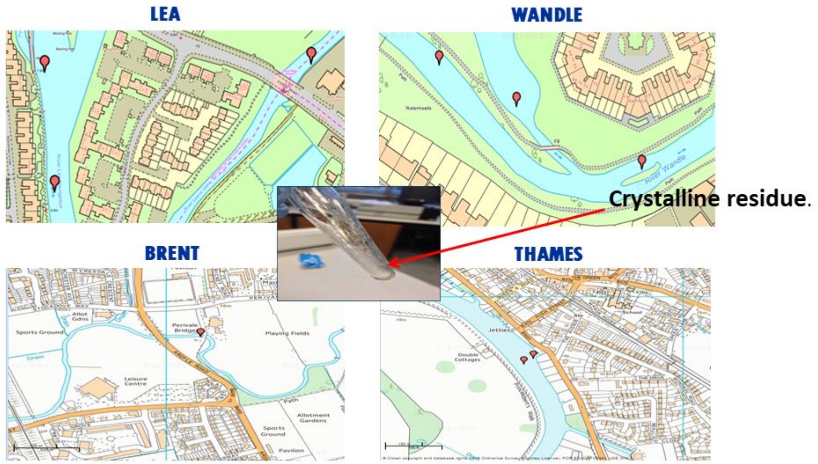




- Analytical Strategies II:
  Environmental Pollution monitoring.

Air, soil, water pollution
Sampling.
Complex sample matrices.
Remote sensing.
Sensor networks.
Data processing.

Studies of London rivers



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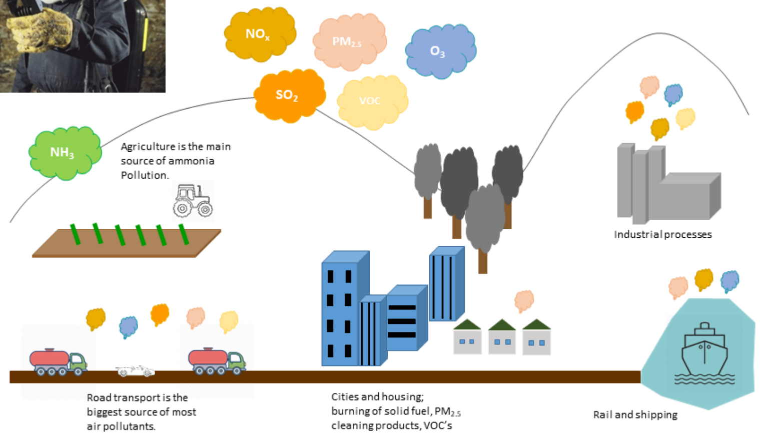

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- Masters level research projects:

  Contamination of waste and surface waters with phenylenediamines from hair dyes.

  Sampling and quantification of Iron particulates in underground train stations.

  Nanodevices for detecting the destruction of cancer cells.

  Photoelectron spectroscopy study of single molecules in the gas phase.

  Measuring lung microbiome in the study of chronic respiratory disease.

- Acknowledgements

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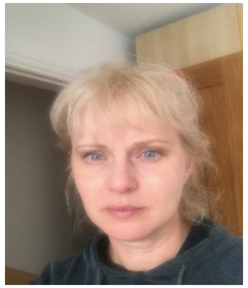

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