MOLS8252

Analytical Measurement Uncertainty and Method Validation

Session 1, Weekday attendance, North Ryde 2020

Department of Molecular Sciences

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General Information

Unit convenor and teaching staff
Lecturer
Danny Wong
danny.wong@mq.edu.au
Contact via (02) 9850 8300
Room 235, 4 Wally's Walk

Credit points
10

Prerequisites
Admission to GradDipBiotech or GradCertLabAQMgt or GradDipLabAQMgt or MBiotech or MBioBus or MLabAQMgt or MRadiopharmSc or MSc or MScInnovationChemBiomolecularSc

Corequisites

Co-badged status
MOLS7252

Unit description
Chemical measurements are required in forensic science, local and international trade, manufacture and production, government regulatory agencies, biotechnology, and nearly every field of science. However, there are always uncertainties associated with measurements owing to experimental errors. This unit systematically covers the estimation principles of measurement uncertainty of values deriving from analytical chemistry measurement procedures and a logical approach to the process of validating an analytical chemistry measurement method. These will then be applied to specific examples from common analytical chemistry.

Important Academic Dates

Information about important academic dates including deadlines for withdrawing from units are available at https://www.mq.edu.au/study/calendar-of-dates

Learning Outcomes

On successful completion of this unit, you will be able to:

ULO1: Interpret the concept of making valid analytical measurements

ULO2: Identify and then evaluate analytical method performance characteristics

ULO4: Deconstruct an analytical method in order to identify factors that influence the

final result

ULO3: Describe the requirements for valid measurements with respect to international standards

ULO5: Evaluate the validity of chemical data using commonly applied statistical techniques

Assessment Tasks

Coronavirus (COVID-19) Update

Assessment details are no longer provided here as a result of changes due to the Coronavirus (COVID-19) pandemic.

Students should consult iLearn for revised unit information.

Find out more about the Coronavirus (COVID-19) and potential impacts on staff and students

General Assessment Information

In order to complete this unit satisfactorily students must

1. submit satisfactory efforts at 3 assignments;
2. submit a satisfactory PowerPoint presentation file for a project;
3. perform satisfactorily in a final examination of three hours duration.

Delivery and Resources

Coronavirus (COVID-19) Update

Any references to on-campus delivery below may no longer be relevant due to COVID-19.

Please check here for updated delivery information: https://ask.mq.edu.au/account/pub/display/unit_status

Technology Used

It is important that you have a scientific calculator as hand-held calculators will be used for assignments and in the final examination. Note that text retrieval calculators are not allowed in the final examination.

Use will be made of Excel and other data processing and display software. Computers carrying this software are available in the teaching laboratories. Items of interest, links to other on-line material will be placed on the unit website.

Prescribed text

J.N.Miller, J,C.Miller, R.D.Miller, *Statistics and Chemometrics for Analytical Chemistry*,

7th Edition, Pearson Prentice Hall, 2018 (ISBN: 978-1-292-18671-9)

Recommended references

D.C.Montgomery, *Design and Analysis of Experiments*, 6th Edition, John Wiley & Sons, 2005 (ISBN: 0 471 48735 X)

R.G.Brereton, *Applied Chemometrics for Scientists*, John Wiley & Sons, 2007 (ISBN: 978 0 470 01686 2)

J.Lawson, J.Erjavec, *Modern Statistics for Engineering and Quality Improvement*, Duxbury Thomson Learning, 2001 (ISBN: 0 534 19050 2)

Eurachem/CITAC Guide: Traceability in Chemical Measurement, Eurachem and CITAC, 2003

ISO/IEC International Standard 17025 General Requirements for the competence of testing and calibration laboratories, ISO, 2005

In House Method Validation: A guide for Chemical Laboratories, LGC Ltd, 2003

Eurachem Guide: the Fitness for purpose of analytical methods, LGC Ltd, 1988

Eurachem/CITAC Guide CG4: Quantifying Uncertainty in Analytical Measurement 2nd Edition, Eurachem & CITAC, 2000

L.Kirkup, *Data Analysis with Excel: An introduction for physical scientists*, Cambridge University Press, 2002

D.B.Hibbert, *Quality Assurance for the Analytical Chemistry Laboratory*, Oxford University Press, 2007

Useful websites

Eurachem -http://www.eurachem.org/

NIST/SEMATECH Engineering Statistics Handbook http://www.itl.nist.gov/div898/handbook/index.htm

Valid Analytical Measurements http://www.vam.org.uk/home.asp

CITAC - http://www.citac.cc/

AOAC - http://www.aoac.org/

NATA - http://www.nata.com.au/publication-updates and download “Technical Note 17”.

Unit Schedule

Coronavirus (COVID-19) Update

The unit schedule/topics and any references to on-campus delivery below may no longer be relevant due to COVID-19. Please consult iLearn for latest details, and check here for updated delivery information: https://ask.mq.edu.au/account/pub/display/unit_status

Proposed schedule

Week 1 + 2	• Statistics
	• Samples and populations
	• Standard deviation of the mean
	• Trueness and precision
	• Significance testing
	  ◦ *t*-test for two means
	  ◦ *F*-test for two variances
	  ◦ Grubb's test for outliers
	  ◦ c2 test
	• Confidence intervals
	• Degrees of freedom
	• Calibration and regression
	  ◦ The correlation coefficient
	  ◦ Standard error of the slope
	  ◦ Standard error of the intercept
	  ◦ Standard error of prediction
	• Basic ANOVA, Two-way ANOVA

Week 3	• Non-linear calibration
	  ◦ Procedure
	  ◦ Standard error of prediction
	• What is validation?
	• What is measurement uncertainty?
	• The importance of analytical measurements
	• What makes a method valid?
	• Fitness for purpose
	• Statistical control (control /Shewhart / CuSum charts)

Week 4	• Review of documents related to validation
	  ◦ ISO 17025
	  ◦ IUPAC technical report 2002
	  ◦ ICH guidelines 2005
	  ◦ Joint AOAC/FAO/IAEA/IUPAC food standard programme
	• ISO 17025 and validation
	• ISO 17025 and NATA
	• IUPAC technical report and validation
	• AOAC / FAO / IAEA / IUPAC and validation
	• ICH guidelines on validation
	• Other documents on validation
	• Verification

Week 5	• Method validation as part of method development
	• Determination of performance characteristics
	• Key performance characteristics
	  ◦ Confirmation of identity
	  ◦ Selectivity
	  ◦ Limit of detection
	  ◦ Limit of quantification
	  ◦ Working and linear range
	  ◦ Sensitivity
	  ◦ Accuracy
	  ◦ Precision – Repeatability and Reproducibility
	  ◦ Recovery

Week 6 + 8	• Further validation
	  ◦ Equipment calibration
	  ◦ Quality control checks, Shewhart control chart
	  ◦ Ruggedness and Robustness
	• Ruggedness testing and experiment designs
	• Factorial designs
	  ◦ 22 factorial designs
	  ◦ 23 factorial designs

	• Optimisation experiments • Response surface concepts and methods • Central composite designs • Box-Behnken designs • Graphical interpretation of response surfaces • Simplex designs
	•
Week 9	• Measurement uncertainty • Why is measurement uncertainty important? • The ISO GUM • The uncertainty estimation process • Specification of a measurand • Sources of uncertainties • Types of uncertainties Cause and effect diagram for use in measurement uncertainty estimation

Week 10	• Quantifying uncertainties
	• Converting uncertainties to standard uncertainties
	• Combining uncertainties
	  ◦ Mathematical expression for the propagation of uncertainties
	  ◦ Rules for combining uncertainties
	  ◦ Sensitivity coefficients

Week 11	• Expanded uncertainties
	• Coverage factors
	• Degrees of freedom
	• The Welch-Satterthwaite equation
	  ◦ Reporting results
	  ◦ Client education
	  ◦ Tools for uncertainty education – The spreadsheet method of measurement uncertainty estimation
	• Measurement uncertainty from a calibration plot

Week 12	• Traceability • Why is it important? • Stated references: SI units • Traceability and uncertainty Measurement uncertainty - a worked example from start to finish
Week 13	• Bias and measurement uncertainty • Other approaches to measurement uncertainty • Bottom up methods • Top down methods • Inter-laboratory studies • Verification of standard methods

Policies and Procedures

Macquarie University policies and procedures are accessible from Policy Central (https://staff.mq.edu.au/work/strategy-planning-and-governance/university-policies-and-procedures/policy-central). Students should be aware of the following policies in particular with regard to Learning and Teaching:

- Academic Appeals Policy
- Academic Integrity Policy
- Academic Progression Policy
- Assessment Policy
- Fitness to Practice Procedure
- Grade Appeal Policy
- Complaint Management Procedure for Students and Members of the Public
- Special Consideration Policy *(**Note:** The Special Consideration Policy is effective from 4 December 2017 and replaces the Disruption to Studies Policy.)*

Students seeking more policy resources can visit the Student Policy Gateway (https://students.mq.edu.au/support/study/student-policy-gateway). It is your one-stop-shop for the key policies you need to know about throughout your undergraduate student journey.

If you would like to see all the policies relevant to Learning and Teaching visit Policy Central (https://staff.mq.edu.au/work/strategy-planning-and-governance/university-policies-and-procedures/policy-central).

Student Code of Conduct

Macquarie University students have a responsibility to be familiar with the Student Code of Conduct: https://students.mq.edu.au/study/getting-started/student-conduct

Results

Results published on platform other than eStudent, (eg. iLearn, Coursera etc.) or released directly by your Unit Convenor, are not confirmed as they are subject to final approval by the University. Once approved, final results will be sent to your student email address and will be made available in eStudent. For more information visit ask.mq.edu.au or if you are a Global MBA student contact globalmba.support@mq.edu.au

Student Support

Macquarie University provides a range of support services for students. For details, visit http://students.mq.edu.au/support/

Learning Skills

Learning Skills (mq.edu.au/learningskills) provides academic writing resources and study strategies to help you improve your marks and take control of your study.

- Getting help with your assignment
- Workshops
- StudyWise
- Academic Integrity Module

The Library provides online and face to face support to help you find and use relevant information resources.

- Subject and Research Guides
- Ask a Librarian

Student Services and Support

Students with a disability are encouraged to contact the Disability Service who can provide appropriate help with any issues that arise during their studies.

Student Enquiries

For all student enquiries, visit Student Connect at ask.mq.edu.au

If you are a Global MBA student contact globalmba.support@mq.edu.au

IT Help

For help with University computer systems and technology, visit http://www.mq.edu.au/about_us/offices_and_units/information_technology/help/.

When using the University's IT, you must adhere to the Acceptable Use of IT Resources Policy. The policy applies to all who connect to the MQ network including students.