



CBMS760

Analytical Measurement Uncertainty and Method Validation

S1 Evening 2014

Chemistry and Biomolecular Sciences

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General Information

Unit convenor and teaching staff

Unit Convenor

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F7B 235

Credit points

4

Prerequisites

Admission to MRes

Corequisites

Co-badged status

CBMS760 is co-badged with CBMS860.

Unit description

This unit covers the estimation principles of measurement uncertainty of values deriving from analytical chemistry measurement procedures and a systematic 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:

Sound interpretation of results after applying appropriate statistical tests to analysis of chemical data

Design method validation studies according to requirements by international standards

Construct an uncertainty budget for an analytical method

Use and evaluate data from methods validated by collaborative trials

Apply the process of deconstructing method so that factors that influence final result can be identified

Assessment Tasks

Name	Weighting	Due
<u>Assignments</u>	30%	To be decided
<u>Project</u>	15%	To be decided
<u>Mid-Year 3-hour Examination</u>	55%	June 214

Assignments

Due: **To be decided**

Weighting: **30%**

On successful completion you will be able to:

- Sound interpretation of results after applying appropriate statistical tests to analysis of chemical data
- Design method validation studies according to requirements by international standards
- Construct an uncertainty budget for an analytical method
- Use and evaluate data from methods validated by collaborative trials
- Apply the process of deconstructing method so that factors that influence final result can be identified

Project

Due: **To be decided**

Weighting: **15%**

On successful completion you will be able to:

- Sound interpretation of results after applying appropriate statistical tests to analysis of chemical data
- Design method validation studies according to requirements by international standards
- Construct an uncertainty budget for an analytical method
- Use and evaluate data from methods validated by collaborative trials
- Apply the process of deconstructing method so that factors that influence final result can be identified

Mid-Year 3-hour Examination

Due: **June 214**

Weighting: **55%**

On successful completion you will be able to:

- Sound interpretation of results after applying appropriate statistical tests to analysis of chemical data
- Design method validation studies according to requirements by international standards
- Construct an uncertainty budget for an analytical method
- Use and evaluate data from methods validated by collaborative trials
- Apply the process of deconstructing method so that factors that influence final result can be identified

Delivery and Resources

• Timetable: Please check <http://www.timetables.mq.edu.au/> for the official timetable of the unit.

• Lectures: The material presented in the lectures is important and you should not assume that all examinable material is available in the textbook or in printed notes. On the other hand, do not assume that all examinable material is to be found in the lecture notes.

• Tutorial: A weekly one-hour tutorial session will immediately follow the lectures.

• Laboratory Work: There is no laboratory work in this unit.

Lecture materials are located in the website for CBMS860 at <https://ilearn.mq.edu.au>.

Macquarie University provides a range of Academic Student Support Services. Details of these services can be accessed at <http://www.students.mq.edu.au>.

Unit Schedule

Week 1 + 2 • Statistics

- Samples and populations
- Standard deviation of the mean
- Trueness and precision
- Significance testing
 - o t-test for two means
 - o F-test for two variances
 - o Grubb's test for outliers
 - o χ^2 test
- Confidence intervals
- Degrees of freedom
- Calibration and regression

- oThe correlation coefficient
- oStandard error of the slope
- oStandard error of the intercept
- oStandard error of prediction
- Basic ANOVA, Two-way ANOVA

Week 3•Non-linear calibration

- oProcedure
- oStandard 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

Week 4•Review of documents related to validation

- oISO 17025
- oIUPAC technical report 2002
- oICH guidelines 2005
- oJoint 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
- oConfirmation of identity
- oSelectivity
- oLimit of detection
- oLimit of quantification
- oWorking and linear range
- oSensitivity
- oAccuracy
- oPrecision – Repeatability and Reproducibility
- oRecovery

Week 6•Further validation

- oEquipment calibration
- oQuality control checks, Shewhart control chart
- oRuggedness and Robustness
- Ruggedness testing and experiment designs
- Factorial designs
- o2² factorial designs
- o2³ factorial designs
- oFractional factorial designs
- oThe Plackett-Burman designs

Week 7•Optimisation experiments

- oResponse surface concepts and methods
- oCentral composite designs
- oGraphical interpretation of response surfaces
- oSimplex designs

Week 8•Multivariate data analysis

- oPrincipal components analysis

- oBackground
- oScores and loadings
- oAlgorithm
- oGraphical representation

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
- oMathematical expression for the propagation of uncertainties
- oRules for combining uncertainties
- Sensitivity coefficients

Week 11•Expanded uncertainties

- Coverage factors
- Degrees of freedom
- oThe 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

oWhy is it important?

oStated 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

oBottom up methods

oTop down methods

•Inter-laboratory studies

•Verification of standard methods

Prescribed text:

J.N.Miller, J.C.Miller, *Statistics and Chemometrics for Analytical Chemistry*, 5th Edition, Pearson Prentice Hall, 2005 (ISBN: 0 131 29192 0)

Recommended references (all available in University Library)

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

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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/>

Policies and Procedures

Macquarie University policies and procedures are accessible from [Policy Central](#). Students should be aware of the following policies in particular with regard to Learning and Teaching:

Academic Honesty Policy http://mq.edu.au/policy/docs/academic_honesty/policy.html

Assessment Policy <http://mq.edu.au/policy/docs/assessment/policy.html>

Grading Policy <http://mq.edu.au/policy/docs/grading/policy.html>

Grade Appeal Policy <http://mq.edu.au/policy/docs/gradeappeal/policy.html>

Grievance Management Policy http://mq.edu.au/policy/docs/grievance_management/policy.html

Disruption to Studies Policy http://www.mq.edu.au/policy/docs/disruption_studies/policy.html *The Disruption to Studies Policy is effective from March 3 2014 and replaces the Special Consideration Policy.*

In addition, a number of other policies can be found in the [Learning and Teaching Category](#) of 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/support/student_conduct/

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 improve your marks and take control of your study.

- [Workshops](#)
- [StudyWise](#)
- [Academic Integrity Module for Students](#)
- [Ask a Learning Adviser](#)

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

IT Help

For help with University computer systems and technology, visit <http://informatics.mq.edu.au/help/>.

When using the University's IT, you must adhere to the [Acceptable Use Policy](#). The policy applies to all who connect to the MQ network including students.

Changes since last offering

There are no changes since CBMS760 was offered in 2013.

Technology Used

It is important that you have a scientific calculator as hand-held calculators will be used during laboratory sessions, 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 unitwebsite.

Computers for general use are provided by the University, but it would be advantageous to have your own computer and internet access.