



# CBMS860

## Analytical Measurement Uncertainty and Method Validation

S1 Evening 2014

*Chemistry and Biomolecular Sciences*

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#### Disclaimer

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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 MLabQAMgt or PGDipLabQAMgt or PGCertLabQAMgt or MSc

Corequisites

Co-badged status

CBMS860 is co-badged with CBMS760.

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 understand data from methods validated by collaborative trials

Understand the process of deconstructing a method so that factors that influence the 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 2014

### 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 understand data from methods validated by collaborative trials
- Understand the process of deconstructing a method so that factors that influence the 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 understand data from methods validated by collaborative trials
- Understand the process of deconstructing a method so that factors that influence the final result can be identified

### Mid-Year 3-hour Examination

Due: **June 2014**

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 understand data from methods validated by collaborative trials
- Understand the process of deconstructing a method so that factors that influence the 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  $\chi^2$  test
- Confidence intervals
- Degrees of freedom
- Calibration and regression
  - o The 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
    - o2<sup>2</sup> factorial designs
    - o2<sup>3</sup> 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 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](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](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](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/](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](http://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](http://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.

## Graduate Capabilities

### PG - Discipline Knowledge and Skills

Our postgraduates will be able to demonstrate a significantly enhanced depth and breadth of knowledge, scholarly understanding, and specific subject content knowledge in their chosen fields.

This graduate capability is supported by:

#### Learning outcomes

- 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 understand data from methods validated by collaborative trials
- Understand the process of deconstructing a method so that factors that influence the final result can be identified

### PG - Critical, Analytical and Integrative Thinking

Our postgraduates will be capable of utilising and reflecting on prior knowledge and experience, of applying higher level critical thinking skills, and of integrating and synthesising learning and knowledge from a range of sources and environments. A characteristic of this form of thinking is the generation of new, professionally oriented knowledge through personal or group-based critique of practice and theory.

This graduate capability is supported by:

#### Learning outcomes

- 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 understand data from methods validated by collaborative trials
- Understand the process of deconstructing a method so that factors that influence the final result can be identified

### PG - Research and Problem Solving Capability

Our postgraduates will be capable of systematic enquiry; able to use research skills to create new knowledge that can be applied to real world issues, or contribute to a field of study or practice to enhance society. They will be capable of creative questioning, problem finding and problem solving.

This graduate capability is supported by:

## Learning outcomes

- 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 understand data from methods validated by collaborative trials
- Understand the process of deconstructing a method so that factors that influence the final result can be identified

## PG - Effective Communication

Our postgraduates will be able to communicate effectively and convey their views to different social, cultural, and professional audiences. They will be able to use a variety of technologically supported media to communicate with empathy using a range of written, spoken or visual formats.

This graduate capability is supported by:

## Learning outcomes

- 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 understand data from methods validated by collaborative trials
- Understand the process of deconstructing a method so that factors that influence the final result can be identified

## PG - Engaged and Responsible, Active and Ethical Citizens

Our postgraduates will be ethically aware and capable of confident transformative action in relation to their professional responsibilities and the wider community. They will have a sense of connectedness with others and country and have a sense of mutual obligation. They will be able to appreciate the impact of their professional roles for social justice and inclusion related to national and global issues

This graduate capability is supported by:

## Learning outcomes

- 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 understand data from methods validated by collaborative trials
- Understand the process of deconstructing a method so that factors that influence the final result can be identified

## PG - Capable of Professional and Personal Judgment and Initiative

Our postgraduates will demonstrate a high standard of discernment and common sense in their professional and personal judgment. They will have the ability to make informed choices and decisions that reflect both the nature of their professional work and their personal perspectives.

This graduate capability is supported by:

### Learning outcomes

- 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
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## Changes since last offering

No changes have been made since the 2013 offering.

## 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 unit website.

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