CBMS760

Analytical Measurement Uncertainty and Method Validation

S1 Evening 2013

Chemistry and Biomolecular Sciences

Contents

General Information

Unit convenor and teaching staff
Unit Convenor
Danny Wong
danny.wong@mq.edu.au
Contact via danny.wong@mq.edu.au
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 understand data from methods validated by collaborative trials

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

Assessment Tasks

Name	Weighting	Due
Mid-year 3-hour Examination	55%	June 2013
Assignments	30%	To be decided
Project	15%	To be decided

Mid-year 3-hour Examination

Due: **June 2013**
Weighting: **55%**

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Assignments

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Weighting: **30%**

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Project

Due: **To be decided**
Weighting: **15%**

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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 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

Unit Outline

Week 1 + 2•Statistics

•Samples and populations

•Standard deviation of the mean

•Trueness and precision

•Significance testing

ot-test for two means

oF-test for two variances

oGrubb's test for outliers

oχ2 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

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://www.mq.edu.au/policy/docs/academic_honesty/policy.html

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

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

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

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

Special Consideration Policy http://www.mq.edu.au/policy/docs/special_consideration/policy.html

In addition, a number of other policies can be found in the Learning and Teaching Category of Policy Central.

Student Support

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

UniWISE provides:

- Online learning resources and academic skills workshops http://www.students.mq.edu.au/support/learning_skills/
- Personal assistance with your learning & study related questions.
- The Learning Help Desk is located in the Library foyer (level 2).
- Online and on-campus orientation events run by Mentors@Macquarie.

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

Details of these services can be accessed at http://www.student.mq.edu.au/ses/.

IT Help

If you wish to receive IT help, we would be glad to assist you at 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 and it outlines what can be done.

Changes since Last Offering

CBMS760 is being offered for the first time 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 unit website.

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