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From Characterisation to Validation: A Journey through Master's Level Analytical Chemistry †

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Abstract: The master's degree in Applied Analytical Chemistry at University College London (UCL) includes valuable teaching input from the UK National Measurement Laboratory for Chemical and Bio-Measurement hosted at LGC. The course starts by introducing accuracy, sensitivity, specificity, trueness, and precision for validating analytical chemistry measurement methods. The principles of proficiency tests, quality control, ruggedness, and associated statistics are practiced using a wide variety of case studies.

Keywords: quality assurance; master's degree; performance parameters; statistics

1. Introduction

Over the years, the increase in chemical measurement has been driven by global industrialisation, instrument affordability, and awareness of the importance and pervasiveness of chemicals to all life. This and an upsurge in citizen science have resulted in many graduates wanting to pursue a career in analytical chemistry [1]. Analytical chemistry is interdisciplinary and applied in many fields of the chemical sciences, agriculture, food, industrial processes, environmental chemistry, pharmaceuticals, fuel, fuel products, cosmetics and forensics, to name a few. It is therefore important to train analytical chemists to evaluate the quality of chemical measurement data. Undergraduate chemists use a sophisticated variety of techniques for chemical characterisation; however, as the emphasis is on the principles and theory of chemistry and physics, this can lead to the impression that data are produced through the art of perfection. Students need to appreciate the complexities of real-world data.

2. Discussion

The MSc degree in Applied Analytical Chemistry at University College London (UCL) seeks to "train the next generation of analytical scientists in state-of-the-art methods. A thorough understanding of uncertainty (error) analysis, data processing and data presentation is at the foundation of this programme." Students start by developing an understanding of what is meant by analytical chemistry and its importance in relation to measurement quality and regulation. The role of chemometrics in analytical chemistry, quantitative univariate statistics, and emerging multivariate data models are included [2]. Understanding performance characteristics in chemical measurement, detection limits, accuracy and bias, precision, reproducibility and ruggedness is crucial to method validation. Chemometrics are therefore used to define measurement performance characteristics in a wide variety of case studies, students consider factors, responses and variables and learn to define these correctly. They then extract analytes from complex matrices and evaluate data from benchtop chromatography and detection instruments. Ranging from quantifying chemical components in perfume, sterols in milk products through to designing sensor systems using Arduinos, as described below. The data are compared with those of their peers and against performance targets. Each stage of the measurement design is evaluated, from sampling techniques and sample variance to sample preparation, extraction, clean up, pre concentration and instrument detection. The impact of variable reduction on improving method ruggedness is addressed with example data and in practice. The contribution to measurement uncertainty is determined at each step, students are introduced to descriptive statistics and the quantitative parameters of uncertainty. Selection of appropriate inferential statistics, Student-*t* tests, general linear models - analysis of variance (ANOVA) or Chi square, is supported by following guidance flow diagrams. An example is comparing two methods for extracting vitamin B9 from Kiwi fruit. Students are also introduced to bootstrapping for model validation, Monte Carlo Simulation and Bayesian conditional probabilities. Quality assessment and quality control systems are taught comprehensively to ensure robust validation of the analytical measurement methods employed. Quality assessment – how this is done, the planned set of activities, Organisation for Standardisation (ISO) and International Electrotechnical Commission (IEC) guidelines and accreditation. Quality control – what is done, the programme of activities, control charts.

A substantial part of the degree course is analytical device construction using using Arduinos (Genuinos), https://www.arduino.cc/, open source electronic prototyping platforms. A sensor development project, as summarised in Figure 1, further enhances skills of critical analysis. This follows known examples, such as the validation of chemo-chronometric assays [3] and use of various optical sensors [4].

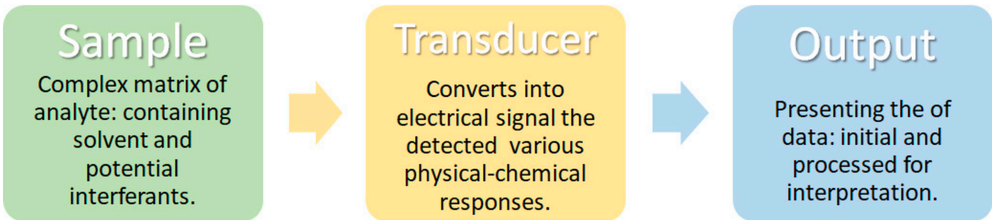

Figure 1. Diagram for manufacturing an analytical device based on Arduino.

Basic electronics, miniaturised detectors, and program coding are used to build sensors, for example, of CO_2 monitoring in lecture theatres, apple ripening, or radioactive decay from Professor William Ramsay's laboratory notebook. The students are supported by a team of analytical method development, electronics and programming experience. A large amount of support resources are also available online. Performance characteristics, processes for validating analytical measurement methods and other inferential statistics tests are used by the students to evaluate their analytical devices. This provides an opportunity to improve their depth of understanding of the importance and impact of the quality of measurement data. Students participate in showcase activities where they demonstrate their devices, discuss and answer questions with fellow students, academic staff and visiting analytical chemistry and chemical measurement expertise. The formal assessment for the analytical device construction is a final report that must include method performance evaluation. This, and a series of inspiring lectures from experts in a range of fields such as environmental analytical chemistry, forensic toxicology, the preservation of historical artifacts, and medical and biochemical diagnostics, lead to various master's level analytical chemistry research projects that embed an understanding of quality assurance. Accuracy, sensitivity, specificity, trueness, and precision for validating analytical chemistry measurement methods, hence principles of proficiency tests are practiced and instilled in the next generation of analytical chemists.

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