Institution: University of Sussex

Unit of Assessment: UoA 05 Biological Sciences

Title of case study: Measurements of contamination sampling

1. Summary of the impact

The chemical contamination of food or soil poses a significant risk to human health; regulatory decisions on the level of this risk are based upon measurements of contamination. To improve these risk assessments, Ramsey devised the 'duplicate method' to estimate the level of uncertainty in measurements of contamination. The application of this method is now included in statutory guidance provided by the soil, food and water sectors to improve reliability in the classification of materials as contaminants and thereby reduce the worldwide risk of contamination to humans.

2. Underpinning research

Regulatory decisions on whether the chemical contamination of food or soil poses a significant risk to human health are based upon measurements of contamination. The reliability of these decisions depends on knowing the full uncertainty of these individual measurements. Previous estimates of this uncertainty only included the contribution from the process of chemical analysis and ignored the often-dominant contribution from the primary sampling process.

New methods were devised by Ramsey to estimate this uncertainty from sampling, including the 'duplicate method'. In the duplicate method, at least eight duplicated samples are taken from around 10 per cent of the sampling targets. This enables the effects of ambiguity in the sampling protocol, and within-target heterogeneity, on the measurement of contaminant concentration to be quantified as uncertainty. Further duplication, for example of the chemical analysis, can also be used to evaluate the dominant contribution to the uncertainty, using statistical interpretation with analysis of variance.

The first description of the 'duplicate method' for the estimation of measurement uncertainty arising from sampling contaminated land was by Ramsey and Argyraki in 1997 [see Section 3, R1] and was later described by Ramsey in 1998 [R2]. The feasibility of this approach for routine site investigation was demonstrated by research conducted for six contrasting sites in 2007 [R3] and, as a result, has since been recommended in recent guidance [see Section 5, C3] by the Environment Agency. Ramsey has also conducted a comparison between this approach and current DEFRA-endorsed procedures that consider just the uncertainty on mean values, based upon multiple samples, rather than the uncertainty of individual measurements [R4].

Key researchers and dates

Ramsey, M.H. [Reader/Professor at Sussex, July 1999–December 2012]

3. References to the research

R1 Ramsey, M.H. and Argyraki, A. (1997) 'Estimation of measurement uncertainty from field sampling: implications for the classification of contaminated land', *Science of the Total Environment*, 198(3): 243–57.

R2 Ramsey, M.H. (1998) 'Sampling as a source of measurement uncertainty: techniques for quantification and comparison with analytical sources', *Journal of Analytical Atomic Spectrometry*, 13(2): 97–104.

R3 Boon, K.A., Ramsey, M.H. and Taylor, P.D. (2007) 'Estimating and optimising measurement uncertainty in environmental monitoring: an example using six contrasting contaminated land investigations', *Geostandards and Geoanalytical Research*, 31(3): 237–49.

R4 Boon, K.A. and Ramsey, M.H. (2010) 'Uncertainty on the measurements of mean value for the reliable classification of contaminated land', *Science of the Total Environment*, 409(2): 423–9.

R5 Ramsey, M.H., Lyn, J.A. and Wood, R. (2001) 'Optimised uncertainty at minimum overall cost to achieve fitness-for-purpose in food analysis', *Analyst*, 126(10): 1777–83.

R6 Taylor, P.D., Ramsey, M.H. and Potts, P.J. (2004) 'Balancing measurement uncertainty against financial benefits: a comparison of *in situ* and *ex situ* analysis of contaminated land', *Environmental Science and Technology*, 38(24): 6824–31.

Outputs can be supplied by the University on request.

Evidence of research quality: peer-reviewed funding

Project title	Funder	Dates	Amount
Assessment of the contribution of sampling total measurement uncertainty estimations	FSA	01/09–12/11	£21k
Cost effective measurement of contamination: sample numbers and uncertainty	EPSRC+Dounreay Site Rest. Ltd	10/09–09/13	£64k + £28k
Quantification of *in situ* heterogeneity of contaminants in soil	EA	10/06–09/09	£6k
Increased acceptability of on-site measurement by estimation and reduction of uncertainty	DTI/TSB	11/06–05/09	£180k
Assessment of practicality and usefulness of sampling proficiency tests in food sector	FSA	10/05–10/07	£160k
Cost-effective investigation of contaminated land	DTI/CL:AIRE	10/03–09/05	£150k
Assessment of optimised uncertainty procedure in practical situations	FSA	05/03–01/06	£211k
A study of measurement uncertainty at limit value concentrations	FSA	01/01–10/03	£33k
Optimised uncertainty at miminum overall cost to achieve fitness-for-purpose in food analysis	FSA	10/99–03/03	£89k
Fitness for purpose of food analysis	MAFF	05/98–02/99	£5k

4. Details of the impact

The first application of the 'duplicate method' to the food sector for estimation of sampling uncertainty was described by Ramsey *et al*. in Sussex in 2001 [see Section 3, R5] and this approach has now been proposed by the Food Standards Agency (FSA) for use in international regulation by the Codex Alimentarius Commission [see Section 5, C4, C5, C6]. This method was then also applied to measurements made on soil *in situ* by Taylor *et al*. in 2004 [R6], and has also now been included in guidance [C3] provided by the Environment Agency.

As a direct result of his published and presented research, Ramsey was approached by a member of the Executive Committee of Eurachem (Alex Williams, also former Government Chemist of the UK), and was commissioned by Eurachem to Chair an international committee for the preparation of the *Eurachem Guide* [C1], which includes case studies across several areas of environmental monitoring including soil, water and food. Eurachem is a network of organisations across Europe and is an independent body that provides a focus for analytical chemistry and quality-related issues, with the objective of establishing a system for the international traceability of chemical measurements and the promotion of good-quality practices (http://www.eurachem.org/). The *Eurachem Guide* describes how to estimate measurement uncertainty arising from sampling using

Ramsey's duplicate method. The *Guide* has been in use throughout the REF period (2008–13) and is widely quoted within the soil, water and food sectors. For example, within the soil sector, this report has informed and modified the guidance on measurements of land contamination set out by the UK Environment Agency and the British Standards Institution (BSI). The UK Environment Agency's guidance on the use of rapid measurement tools at contaminated sites [C3] cites the *Eurachem Guide* [C1], exemplifying the use of the duplicate method and uncertainty information for the probabilistic mapping of soil contamination. The 2011 revision of the British Standard (BS 10175) on the investigation of contaminated land [C2] similarly cites the *Eurachem Guide* [C1], and includes guidance to the contaminated land community in the UK on how to estimate uncertainty from sampling using the duplicate method. Within the food sector, Ramsey's research has also informed Codex, an international body organised by the Food and Agriculture Organization and the World Health Organization (FAO/WHO). Two Codex committees, at three annual meetings between 2008–10 [C4, C5, C6], discussed the role of uncertainty from sampling in the regulation of the international trade of foods. Codex recommend *'The measurement uncertainty of an analytical result including uncertainty from sampling may be estimated by a number of procedures, notably those described by EURACHEM* [C6]*.* In addition, guidance supporting the implementation of the EU Water Framework Directive [C7] now cites the *Eurachem Guide* [C1] and describes the use of the duplicate method.

5. Sources to corroborate the impact

C1 Ramsey, M.H. and Ellison, S.L.R. (eds) (2007) *Measurement Uncertainty Arising from Sampling: A Guide to Methods and Approaches*. *Eurachem/EUROLAB/CITAC/Nordtest/AMC Guide*. Uppsala: Eurachem. http://www.eurachem.org/index.php/publications/guides/musamp

C2 BS 10175:2011 *Investigation of Potentially Contaminated Sites – Code of Practice. Annex D (informative) The Assessment and Control of Sampling Uncertainty*. London: British Standards Institute.

C3 Environment Agency (2009) *Framework for the Use of Rapid Measurement Techniques in the Risk Management of Land Contamination*. Bristol: Environment Agency, Science Report.

C4 Joint FAO/WHO Food Standards Programme Codex Committee on Pesticide Residues, Fortieth Session, Hangzhou, China, 14–19 April 2008. Discussion paper on the estimation of uncertainty of results for the determination of pesticide residues (discussed uncertainty of sampling for pesticide residues, with reference to the *Eurachem UfS Guide* p. 4).

C5 Joint FAO/WHO Food Standards Programme Codex Committee on Methods of Analysis and Sampling, Thirtieth Session, Balatonalmádi, Hungary, 9–13 March 2009, Agenda item 9, Guidance on uncertainty of sampling (prepared by the UK) (Item 104: discussed uncertainty of sampling of food in general in the light of *Eurachem UfS Guide*, pp. 1–18).

C6 Joint FAO/WHO Food Standards Programme Codex Committee on Methods of Analysis and Sampling, Thirty-first Session, Budapest, Hungary, 8–12 March 2010, Agenda item 6, Guidance on uncertainty of sampling (prepared by the UK) (further discussed uncertainty of sampling in the light of *Eurachem UfS Guide*, pp. 1–17, with recommendation on p. 7).

C7 *Common Implementation Strategy for the Water Framework Directive* (2000/60/EC) Guidance Document No. 15: *Guidance on Groundwater Monitoring* (ISBN 92-79-04558-X). Case study 'Estimation of groundwater monitoring uncertainty' (pp. 47–8) available at http://circa.europa.eu/Public/irc/env/wfd/library?l=/framework_directive/guidance_documents/nov-2006_final-2pdf/_EN_1.0_&a=d.

A fuller version of same case study on water is also used as Example A3 in the *Eurachem UfS Guide* (pp. 46–54).

C8 In addition to the above documents, individual end-users/beneficiaries who could be contacted to corroborate this impact include contacts at the Environment Agency and the Food Standards Agency.