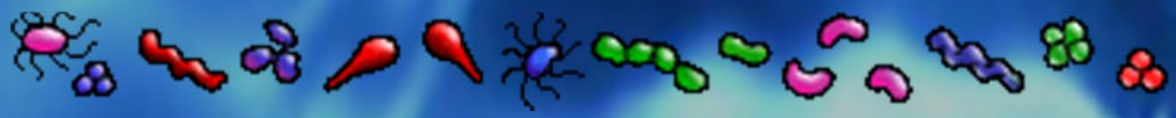
Analytical Quality Control:

Where are we and where do we go?



David Sartory
SWM Consulting

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New River Head Laboratories

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So, what is
Analytical
Quality Control?
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What is Analytical Quality Control ?

- **ISO 9000 - Quality management systems - Fundamentals and vocabulary (2005)**
- **Quality Assurance**

  'part of quality management focused on providing confidence that quality requirements will be fulfilled'

- **Quality Control**

  'part of quality management focused on fulfilling quality requirements'

- **Quality Management**

  'co-ordinated activities to direct and control an organisation with regard to quality'

What is Analytical Quality Control ?

- **ISO 8402 - Quality management and quality assurance - vocabulary (1994)**

- **Quality Assurance**

  'all those planned and systematic actions necessary to provide adequate confidence that a product, process or service will satisfy given quality requirements'

- **Quality Control**

  'the operational techniques and activities that are used to fulfil requirements for quality'

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Basic Analytical Quality Control

- Understanding ALL the factors that can have an impact on results of analyses

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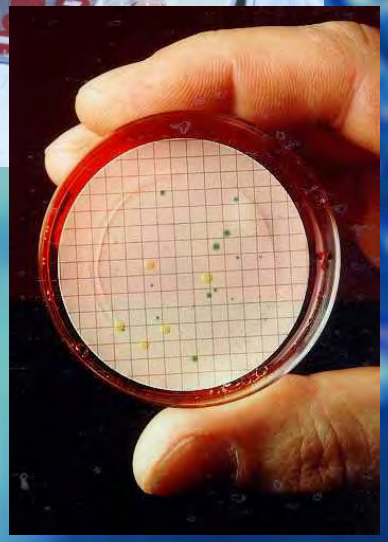
Basic Analytical Quality Control



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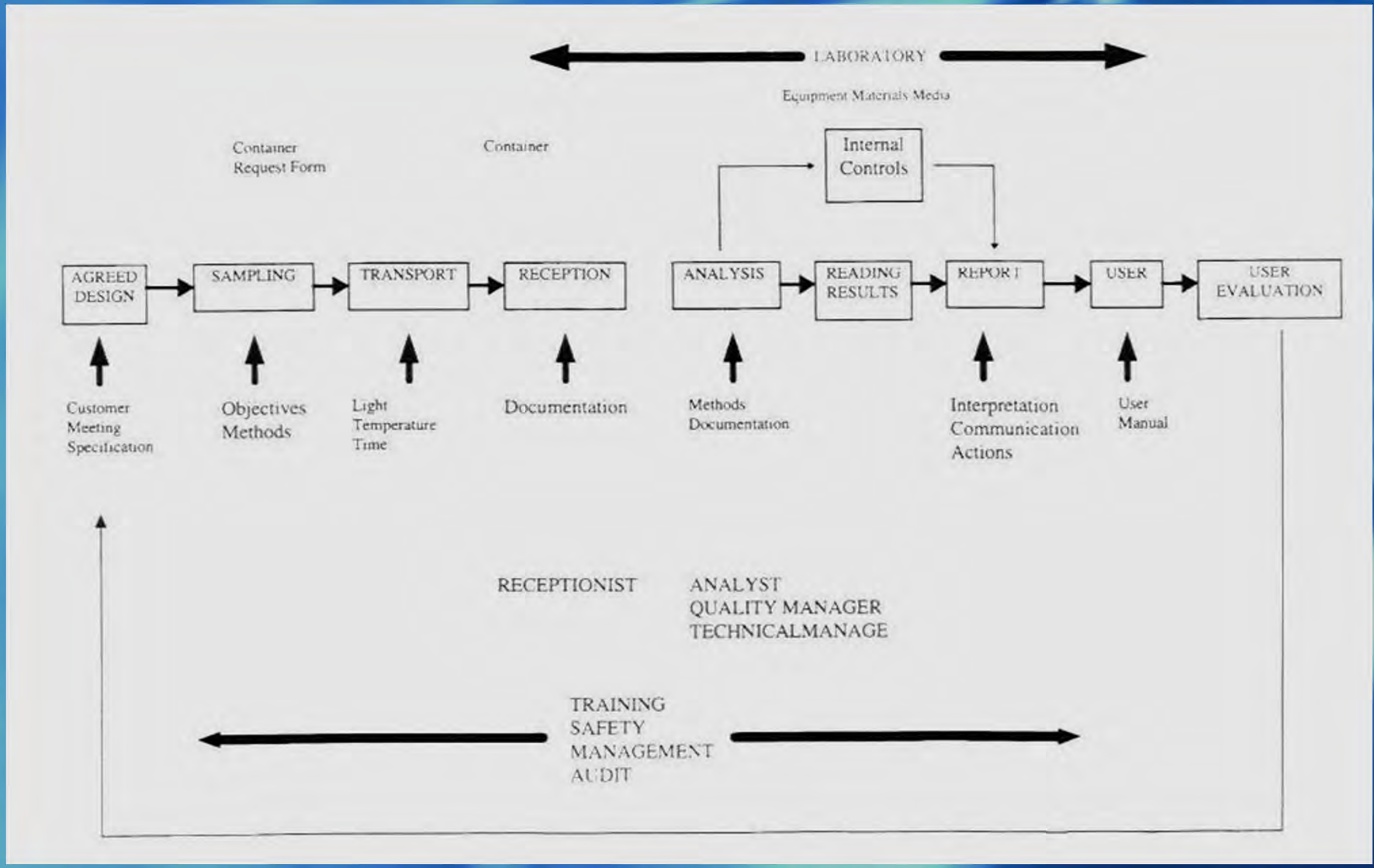
Basic Analytical Quality Control



From Lightfoot, N. F. & Maier, E. A. (Eds.) (1998) Microbiological Analysis of Food and Water - Guidelines for Quality Assurance

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International Guidance

- ISO/IEC 17025:2005 General requirements for the competence of testing and calibration laboratories
- ISO 8199:2005 Water quality – General guidance on the enumeration of micro-organisms by culture
- ISO/DIS 11133:2012 Microbiology of food, animal feeding stuffs, food production environment and water - Preparation, production, storage and performance testing of culture media

National Guidance

- **UKAS guidance**
- ***Microbiology of Drinking Water (2010) - Part 2 - Practices and Procedures for Sampling***
- ***Microbiology of Drinking Water (2002) - Part 3 - Practices and Procedures for Laboratories*** **(currently being revised and substantially expanded)**
- **DWI guidance regarding "competent analysts"**

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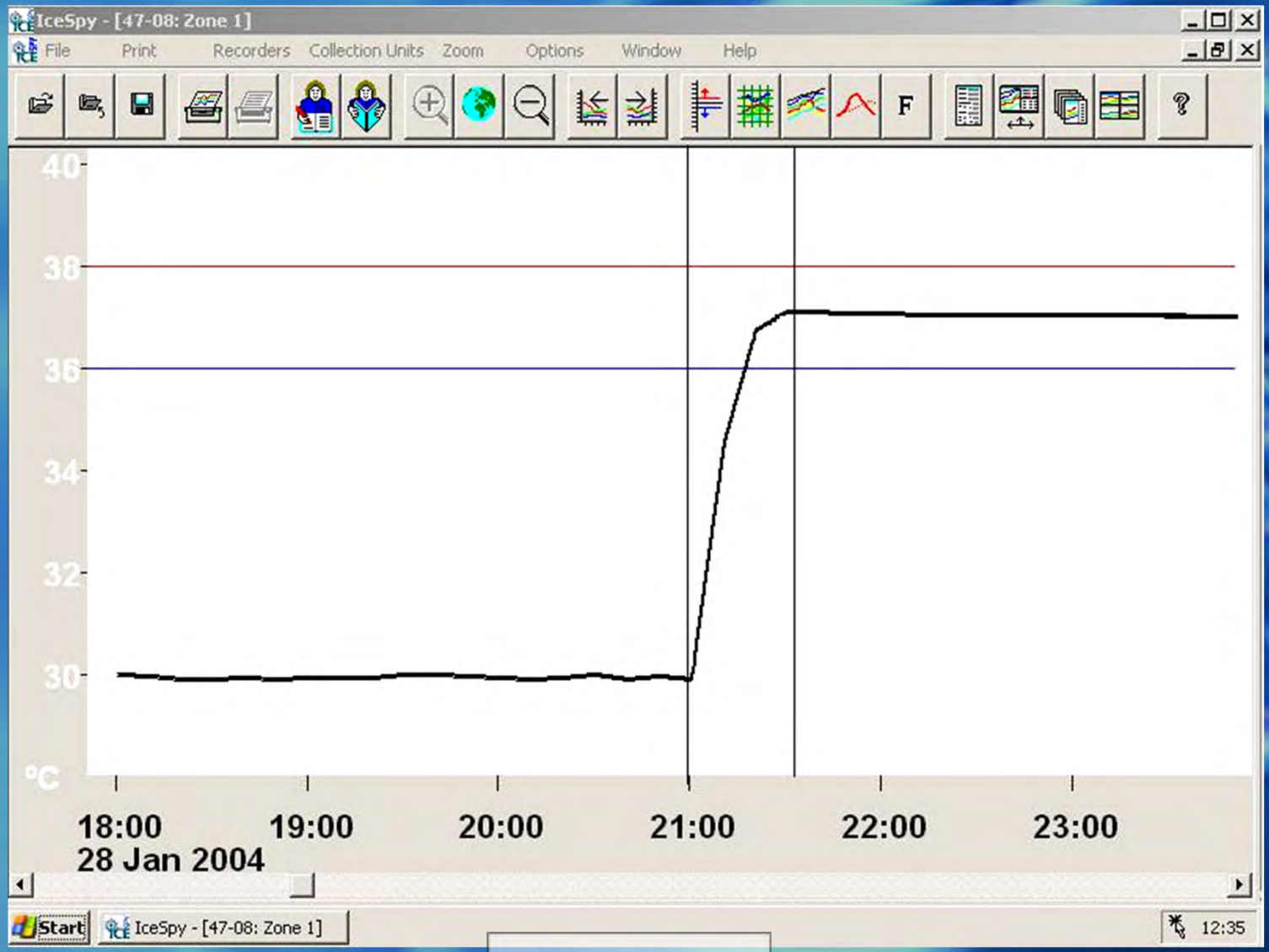
Some Major Changes

- Virtually every aspect of analysis
- ISO 17025 and accreditation !!!
- Regulatory oversight (DWI and other regulators)
- Analyst training and ongoing development
- Much more reliable equipment
- Development of external proficiency schemes
- Development of reliable reference materials
- Availability of quality assured media
- Guidance and framework for adopting new methods


So, some examples

Cycling Incubators



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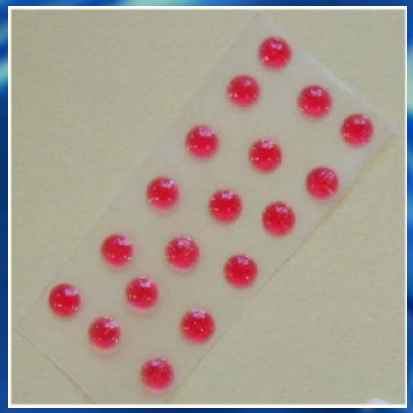
Internal and External Quality Control

- **Internal Quality Control**
  - equipment and microbial growth performance
  - devised and managed by laboratory
  - micro-organisms normally analysed for by laboratory
    - positive, negative and blank controls
    - reference strains or environmental isolates
    - characterised environmental sample
    - HPA Lenticule™
- **External Quality Control**
  - external independent body
  - samples contain micro-organisms in unknown numbers

Internal Control - Reference Materials

- **Lenticule™ (HPA, Colindale Avenue)**

  - 10 to 10^8 cfu per lenticule
  - easy to use
  - representative bacteria



- **BioBall ™ (BTF, Australia)**

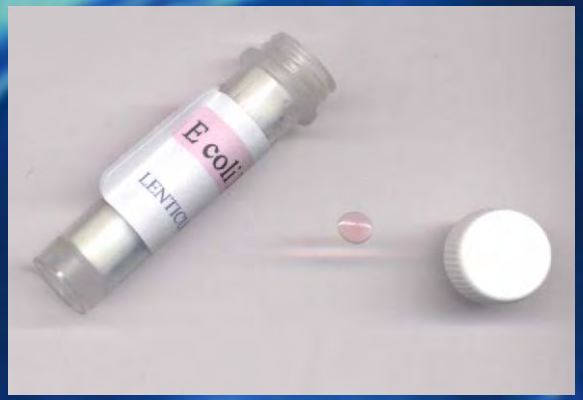
Selectrol® (TSC Biosciences, Buckingham)

Vitroid ™ (CDP, Newcastle)
RapidCheck ™ (RTC Wyoming, USA)

External Quality Control Schemes

HPA EQUAL (Water Microbiology EQA Scheme)

- large participation (over 250 UK laboratories)
- frequent (10 per year)
- Lenticule™ samples
- simulated unchlorinated drinking water
- bathing water (1 per year)
- also *Legionella*



Health Protection Agency
WATER EQA

LEAP Scheme

- Laboratory Environmental Analytical Proficiency
- operated by Central Laboratory Services
- monthly water samples stabilised (2 – 3 days limit)
- water micro and C*ryptosporidium*/*Giardia*
- limited number of participants

Central Science Laboratory

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External Quality Control Schemes

Aquacheck

- organised by QM (+ LGC)
- lypholised samples
- simulated drinking, process and other waters
- limited number of participants

Aquacheck

DWI Crypts Scheme

- organised by LGC and SPDL for DWI
- *Cryptosporidium* only
- oocysts on slides
- suspensions for IMS and staining
- inoculated filters

crypts

SPDL
SCOTTISH PARASITE DIAGNOSTIC LABORATORY

LGC
Setting standards in analytical science

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Understanding
the Statistics!
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Difference with Chemistry

- It is so different from chemistry
- A 1O ml sample containing 50 µg/l aluminium

  Mean number of ions

  = 11 160 118 600 000 000 per sample

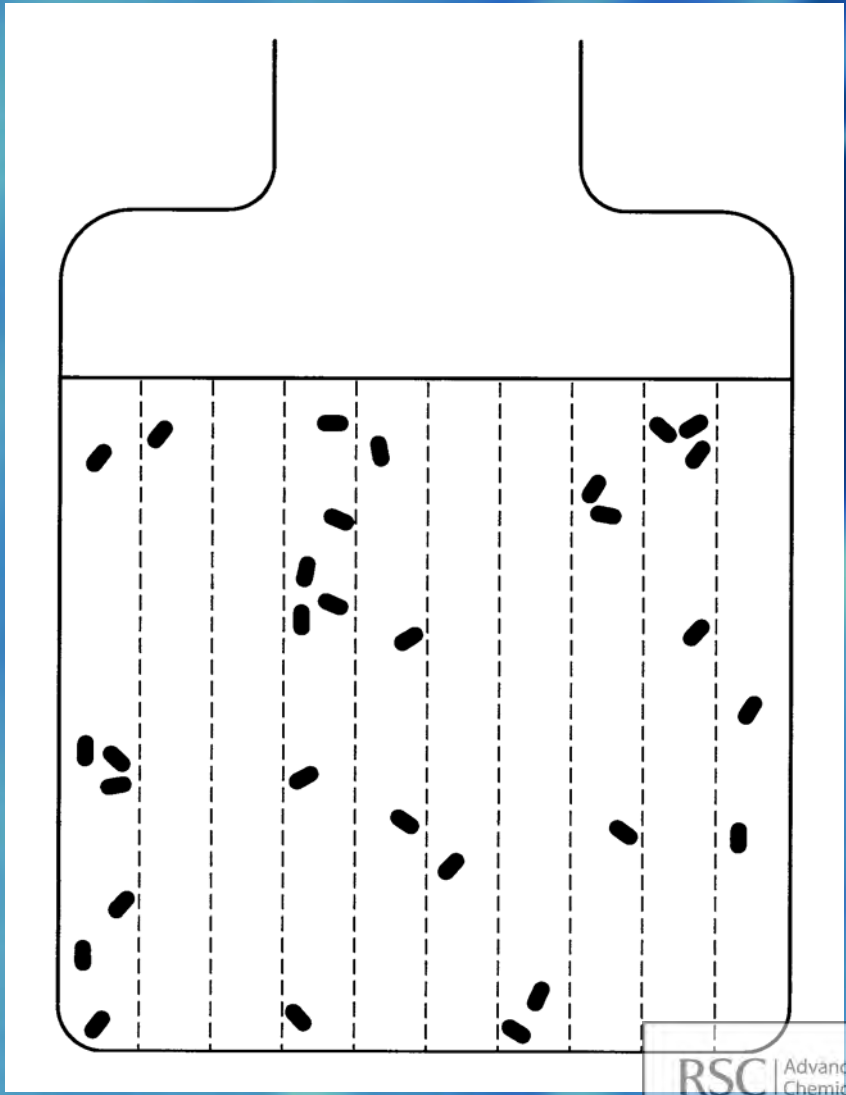
- Assuming random variation then 95 % of samples would contain between:-

  11 160 118 392 942 734 and

  11 160 118 807 572 665 ions

- Since only the first four or five digits will effect the results of analyses, there can be significant natural variation before affecting any results !!

Bacterial Numbers in Water: impact of natural dispersion



30 bacteria distributed at random in a sample, split into 10 equal sub-samples

Dispersed particles not a solution

Counts per sub-sample range from 0 to 7

Image courtesy of BSi

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Uncertainty of Measurement

- Not appropriate for water microbiology, particularly for samples with low counts as found in drinking water
- BS 8496:2007 Water quality – Enumeration of micro-organisms in water samples – Guidance on the estimation of variation of results with particular reference to the contribution of uncertainty of measurement
- ISO/DIS 29201:2011 Water quality - The variability of test results and the uncertainty of measurement of microbiological enumeration methods

Validation, Verification and Adoption of New Methods

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Validation (ISO/TR 13843)

- Derivation of method specifications
  - specifications of method requirements
  - specifications of performance
  - conducted by experienced technical staff
- Unambiguous description of target organism
- Specifications of performance
  - morphological identification
  - incubation conditions and media characteristics
  - limits of numbers detectable (working limits)
  - counting uncertainty
  - sensitivity and selectivity (false-positives/negatives)
  - limitations

Verification: your place or mine?

- Microbiological methods have a nasty habit of working well in some laboratories and not others !!

  → Assess performance of method in your laboratory against specifications

- Simplified primary verification procedures
- Verification of performance
  - confirmation of morphology
  - counting uncertainty
  - confirmation of target and non-target organisms
  - sensitivity and selectivity (false-positives/negatives)
  - analyst variability

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Directives and Development: a case of necessity

- **1998 – European Drinking Water Directive**
  - **for the first time stipulated methods for five microbiological parameters**
  - **ISO standards for *E. coli*, coliforms, enterococci and heterotrophic bacteria counts**
  - **mCP method for *Clostridium perfringens***
- **Alternative methods *"may be used, provided that the results obtained are at least as reliable as those produced by the methods specified"***

Comparison of Methods: protocols and statistics

- 1999 – UK Drinking Water Inspectorate commissioned development of protocols and a robust statistical approach to establishing equivalence
- 2000 – Procedure used to compare UK methods for *E. coli* and coliforms against ISO 9308-1 (incorporated into *The Microbiology of Drinking Water* Part 3 in 2002)
- 2000 to 2004 – ISO 17994 developed refining the UK statistical approach

Basic Requirements of ISO 17994

- Range of samples over wide geographical area
  - appropriate for test normally conducted
  - for drinking water – chlorine stressed if disinfected
- Enough target bacteria for low likelihood of zero counts (target 10 – 30 colonies/plate)
- Sufficient samples with counts to detect an 'average relative difference' of 10 % units
  - 256 samples
- Confirm all presumptive target colonies
- Expert panel, including statistician

Comparing Methods: what is equivalence? – ISO 17994

- **Based on *Relative Difference* calculated from paired data**

$$x_i = [\ln(a_i) - \ln(b_i)] \times 100\ \%$$

where a = test method and b = reference method

- ***Expanded Uncertainty* calculated from the standard deviation of the mean relative difference and a coverage factor (k) of 2**

$$U = 2s / \sqrt{n}$$

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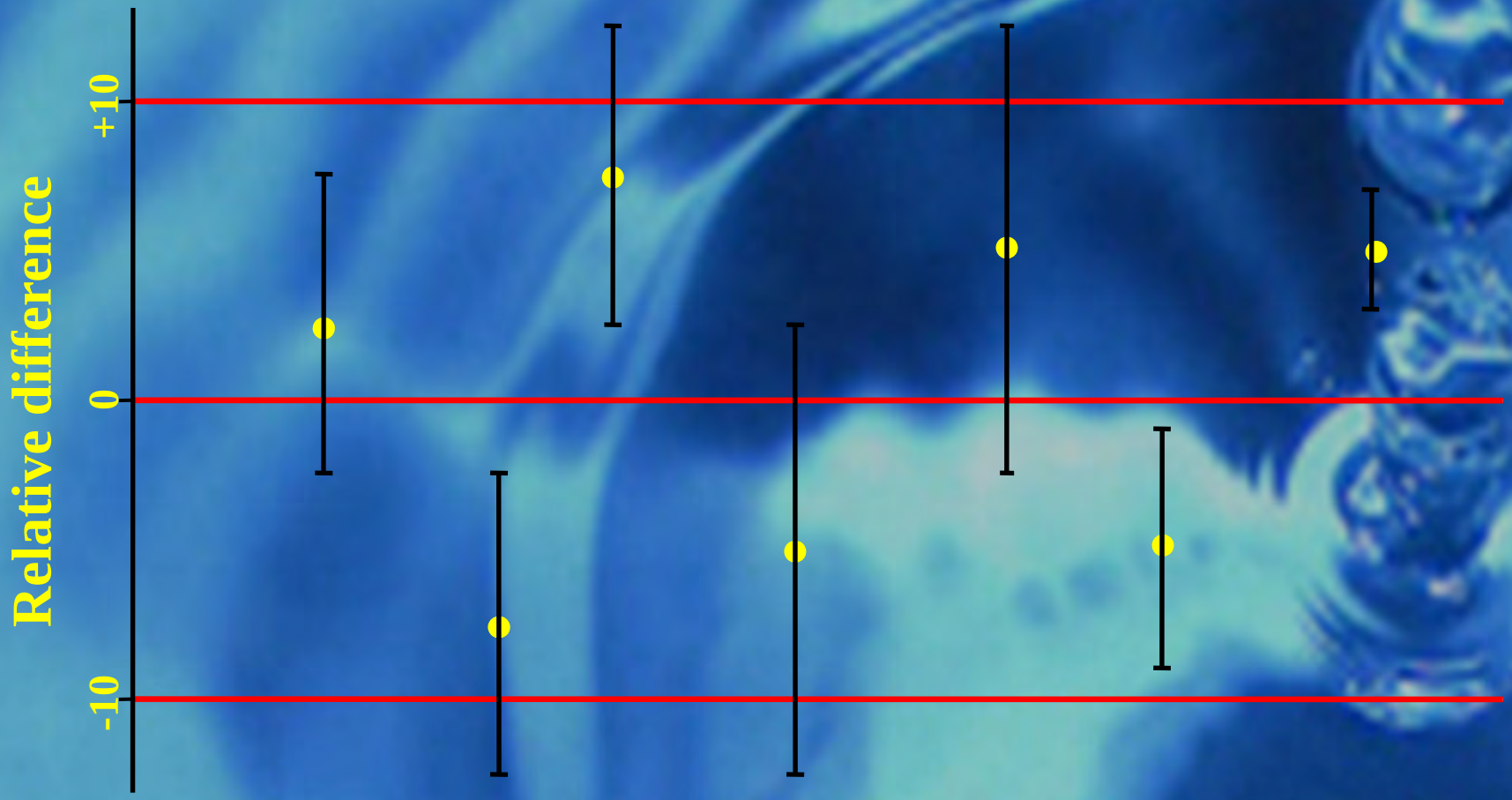
Comparison of Methods



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Thanks to:

- RSC Water Science Forum for the opportunity to sound off!!
- But mostly to the numerous colleagues and fellow travellers over the last 35 years for the debate, enumerable pints of beer, bottles of wine and endless discussions on analytical water microbiology!!

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