Proficiency Testing in a Global Analytical Environment—Benefits & Challenges

AOAC

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The Coca-Cola Company is the largest manufacturer, distributor and marketer of nonalcoholic beverage concentrates and syrups in the world.

Finished beverage products bearing our trademarks, sold in the United States since 1886, are now sold in more than 200 countries.

Along with Coca-Cola, the world’s most valuable brand, we market four of the world’s top five nonalcoholic sparkling brands, Diet Coke, Fanta and Sprite.

Approximately 52 billion beverage servings of all types are consumed worldwide every day — beverages bearing trademarks owned by or licensed to The Coca-Cola Company account for more than 1.4 billion.
The Coca-Cola Company has approximately 71,000 employees in the following operating segments:

- Africa
- East, South Asia and Pacific Rim
- European Union
- Latin America
- North America
- North Asia, Eurasia and Middle East
- Bottling Investments
- Corporate
Assurance Of Analytical Proficiency

- Analyst Certification Program (analytical and microbiological)
- ISO 17025 Accreditation
- Internal Proficiency Testing Programs (ingredients, intermediates and finished goods)
- Laboratory Audits (TCCQS ISO 2000)
- Adoption of industry standard methods (AOAC, USP, FDA, EPA...)
- Formal Method Validation
- External Laboratories Audit and Authorization Process
- Check Sample Programs
- Proficiency Testing, Inter-Laboratory Comparisons
Benefits and Uses of Proficiency Testing

- Benchmark and Demonstrate Technical Capabilities
  - Independent review of results
  - Document performance and capability
  - Improve laboratory skills
  - Training

- Identify analytical issues with sample matrix and analytes of interest
  - To develop sampling and testing programs with scientific proof

- Identify best practices and best laboratories for specific fields of work
  - The importance on method validation expertise cannot be underestimated even when using mandated methods

- Reduce cost by addressing logistic issues that could render the measurements unusable or scientifically flawed
Benefits and Uses of Proficiency Testing

- In many countries, commercial laboratories are required to participate in specific PT programs
  - For each specific field of work (e.g., organics in water)
  - To obtain local/international recognition
  - To demonstrate Technical and Analytical Capabilities
  - To benchmark against best scientific practices

- For example: water testing for environmental discharge or human consumption
  - USA NELAC/ISO 17025 Standards
  - EU IUPAC/ISO/AOAC International Protocol for Proficiency Testing

- To facilitate and promote Free Trade many countries are signatories of international agreements that require PT
  - ILAC
  - APLAC
  - NELAP
Challenges Of Proficiency Testing Programs

CHALLENGES IN PROFICIENCY TESTING

- LOGISTICS
- ANALYTICAL
- DATA INTERPRETATION
Challenges Of Proficiency Testing Programs

LOGISTICS

- Availability of a Commercial PT Program for the Specific Analyte of Interest
- Number of Laboratories Necessary to Conduct a Valid Inter-laboratory Comparison
- Shipping and Handling of Samples
  - Availability of Carriers
  - Spills in Traffic
- Classification of Shipments by Country
  - Corrosive
  - Toxic
- Sample Integrity
  - Customs Opens the Package
  - Repeatability
  - Sample Homogeneity
    - Solubility and Partition
  - Sample Stability
    - Preservation
      - Refrigeration
      - Acidification
      - Addition of Chemicals
- Cost
  - Cost to Prepare the Study
  - Cost to Run the Samples
  - Cost to Ship and Handle the Samples
  - Cost to Interpret and Report Results
Challenges Of Proficiency Testing Programs

- Target Analytes
  - Typical or Special
- Trained Analysts
  - Demonstration of Capabilities
- Reference Methods
  - Detection Limits
    - Influenced by Technology
      - L-L Extractions
      - Solid Phase
    - Mandated or None Available
- Reference Standards
  - Availability
  - Purity
  - Stability
  - Toxicity
- Chain of Custody
  - Sample Mix-up and Incorrect Identification
  - Lost Samples
- Calibration Errors
  - Calculations
  - Preparation
  - Dilutions
Challenges Of Proficiency Testing Programs

Data Interpretation

- True Value vs. Central Tendency or Weighed Value
  - Is True Value Known?
  - Distribution of Results
  - Acceptability
    o Arbitrary
    o Industry
- Uncertainty Is Unknown
- False Positives
  - Sample Contamination
  - Carryover from Spikes
  - Matrix Related
- False Negatives
  - Sensitivity
  - Identity
  - RL
As water is our highest volume ingredient, it is necessary to have state-of-the-art internal and external capabilities to assess and monitor safety and quality at all times.

- For all regulated compounds, we employ the best available technology
- Mandated methods are adopted when available (i.e. EPA, FDA…)
- Modifications are sometimes necessary to achieve lowest detection limits as per our global standards
- We benchmark our laboratories against the best in class for each field of work
Study Overview

- A major PT provider was contracted to conduct a complete assessment on the technical capabilities for water testing on reference laboratories
  - USA (2), India (1), Europe (2)
  - Each laboratory was required to analyze water samples spiked with known concentrations of the following target analytes
    - Volatile Organics (6 analytes)
    - Trihalomethanes (4)
    - Pesticides/Semivolatiles (15)
    - Carbamates (4)
    - Herbicides (6)
    - Metals (7)
    - Inorganic Disinfection Byproducts (3)
    - Nitrate (1)
Proficiency Testing to Determine Analytical Capabilities in Water Testing

Study Overview

- **Samples:**
  - Blank (1)
  - raw Water (4)
    - 1 ppm Cl₂ residual
    - Preserved and unpreserved
  - Treated water (2)
    - Preserved

- Analyze levels above and below RL’s

- **Testing Schedules**
  - Day 1, 3, 14, 21

- **Methods**
  - VOAs & THMs
    - USA1, USA2, and India – 524.2 (Purge/Trap GC-MS)
    - EU1 and EU 2 – Headspace-GC-MS
    - EU2 – Headspace-GC/ECD for THMs
  - Pesticides/Semivolatiles
    - USA1, USA2, India – 525.2 (Liquid/Solid Extraction-GC-MS)
    - USA1 – 7 Pests. by 505 (Microextraction-GC)
    - EU1 – SPE/GC-MS
    - EU2 – SPE-GC-MS
Study Challenges Logistics

Logistics

- Complex Study Scheme –
  - Different sample fragments timed to assess sample stability and preservation effectiveness
  - Laboratories in different countries
- Different carriers were needed
  - Samples to USA and Europe delivered next day
  - Shipping to India – two weeks, first shipment confiscated at customs. We sent a second shipment through a different carrier that got through
  - Shipment labeled as “corrosive”
- Complex preservation scheme
  - Refrigerated
  - Chemical preservation
  - Blanks, controls and checks
- Cost of preparation, shipping and analysis of results > 150,000
GC Pesticides data are more accurate with lower RSDs than GC-MS
  • Better sensitivity

EU2 LC-MS Dimethoate data better than GC-MS of others
  • Proprietary method outperforms the mandated method?

India’s AA metals data worse than ICP/MS of others
  • Outside of distribution
  • Issues with the acidification

Systematic Calibration Errors Observable for EU1 Lab
  • Investigation necessary to validate their results
  • Dilution error yielded reported results 10X greater than true value

Many False Negatives
  • suggesting their advertised DL’s are may not be scientifically derived
  • Analyte Identification and confirmation practices?
Study Learnings

- Your results will only be as good as your sampling practices
- Methodology differences may not be critical
  - Good data was obtained for all methods when properly executed
- External Certifications and Accreditations do not seem to have impact over the quality of the data (and in many cases there could be misleading)
- Laboratories that perform the test routinely did not do better than those that do it with less frequency
  - Discipline and capabilities are important
- Communication between the laboratory and the Company is always critical in minimizing errors and explaining deviations
- Preservation is most cases makes a big difference in sample integrity
  - Support literature that preservation is necessary at time of collection
  - Best preservation technique is refrigeration, followed by the chemical preservation
  - Metals and nitrate are stable
  - Unpreserved samples yield false negatives!
- It is money well spent
  - If you select the right PT provider and the right laboratories
In most situations, the laboratory knows the sample is artificial
- They are alerted of the upcoming test

The spikes are normally too high and do not challenge the DL’s

Analysts can repeat the test and provide averaged data

Samples are clean and unnatural — artifacts such as other contaminants are not represent to challenge the selectivity of the methods

Recoveries are high due to the concentration

Laboratories may assign the PT sample to their best analyst but your typical sample goes to the average analyst
Thank You
Effect of Sample Preservation Technique in Sample Stability

Heptachlor Stability

Days Upon Preparation

conc (µg/L)

Preserved

Unpreserved