Laboratory Control Sample (LCS) Study Overview

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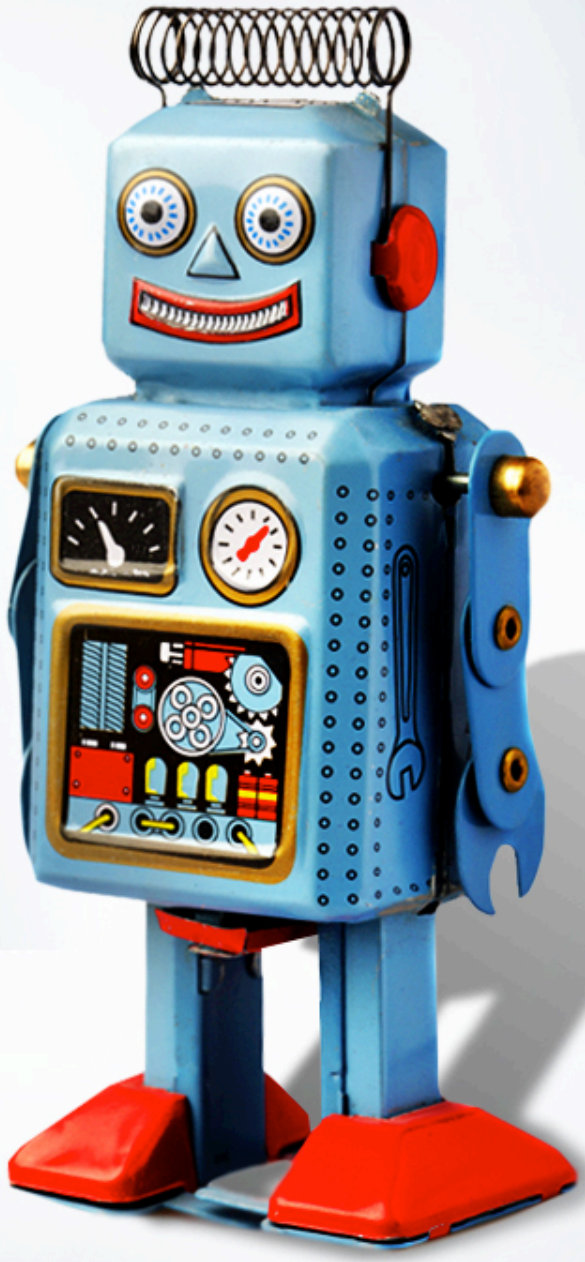

Agenda

- **Fred McLean (Navy)** - Overview of the Laboratory Control Sample (LCS) Study
- **Kelly Black (Neptune)** – The statistics behind the study
- **Chris Gunning (A2LA)** – How an Accrediting Body (AB) will assess the laboratory for compliance with the QSM Control Limits Tables
- **Charles Stoner (Army Public Health)** – How laboratories are integrating the LCS results
- **Kari Meier (Army Corps)** – How projects can integrate this study into their QAPPs

Old vs. New: The Original Study

1999 LCS Study: Data was collected in 1999; results were published in the 2004 LCS Study report

20 of the "best" DoD approved labs were asked to participate

Most methods had a maximum of 17 labs send data, with 5 labs considered a minimum

Labs were asked to send their last 20 LCS values for each method/analyte without censoring

The Original Study (2004)

Control Limits were set at 3 standard deviations around the mean for all methods

Method 8151 (Herbicides) control limits were considered "too wide" for use; the 5th and 95th percentiles were used instead

Control Limits were rounded to the nearest 5 for "ease of use"

Lower Control Limits were raised to a minimum of 10

Original Study Goals

Replace AFCEE Control Limits with empirical data collected in cooperation with American council of Independent Laboratories (ACIL)

Purpose: To evaluate how well the more commonly used SW-846 methods performed when conducted in routine fashion. 454 analyte-matrix-method combinations over 9 methods were collected and analyzed

Use the limits as a set of "benchmarks" that labs were required to achieve, regardless of method parameters. Limits were expected to be used in comparison with new technology

All analytes spiked in the LCS shall meet the DoD generated limits (for lab approval)

LCS Study Overview
2004 Study

Methods Evaluated:

- 6010 – Metals/ICP-AES
- 7470/7471 – Mercury/CVAA
- 8081 – Organochlorine Pesticides/GC
- 8082 – PCB Aroclors/GC
- 8151 – Chlorinated Herbicides/GC
- 8260 – Volatile Organics/GC-MS
- 8270 – Semi-volatile Organics/GC-MS
- 8310 – PAHs/HPLC
- 8330/8330A – Explosives/HPLC

LCS Study Overview: 2013

- Purpose: To re-evaluate laboratory performance using a larger and more recent data set and update limits published in the DoD QSM
- Parameters: 23 methods, 52 laboratories, 1258 analyte-matrix-method combinations
- Participating laboratories were either accredited in accordance with DoD ELAP or assessed in accordance with DOE-CAP
- Approximately 6.6 million records were uploaded, of which 91% were successfully processed and used for setting control limits

2013 LCS Study Parameters

- A minimum of one year's LCS data was collected from each laboratory, representing at least 30 results per analyte/matrix/method combination
- No data older than 3 years accepted
- Data cleaning steps resulted in exclusion of approximately 9% of submitted data

2013 LCS Study

Additional methods evaluated:

SW-846 Methods

- 6020 – Metals - ICP/MS
- 6850 – Perchlorate - HPLC/ESI/MS; MS/MS
- 7196 – Hexavalent Chromium - Colorimetric
- 8015 – Nonhalogenated Organics - GC
- 8141 – Organophosphorus Compounds - GC
- 8270 SIM – Semivolatile Organics - GC/MS
- 8290 – Dioxins/Furans - HRGC/HRMS

2013 LCS Study

Additional methods evaluated:

SW-846 Methods (cont'd.)

- 8321 – Non-volatile Organics – HPLC/TS/MS
- 8330B – Explosives – HPLC/UV
- 9010-9021 – Cyanide - Various
- 9056 – Inorganic Anions - IC

Non-SW-846 Methods

- RSK-175 – Dissolved Gases - GC
- TO-15 – VOCs in Air/Canister - GC/MS
- 1668 (CWA) – PCB congeners - HRGC/ HRMS

2013 LCS Study Results

Based on 1,258 analyte-matrix-method combinations:

Bias (mean LCS recoveries across laboratories)

- 97% of limits centered at ≥ 60%
- 67% of limits centered at ≥ 90%

Precision (range of LCS recoveries across laboratories)

- Only 14% of ranges within ± 20%
- 97% of ranges within ± 75%

How do the results compare with the 2004 study?

The mean LCS percent recovery improved since 2004 for 72% of the limits.

- OK, enough with the statistics. Have control limits gotten "better" or "worse" since the last study?

  Answer: Neither

- Calculated QSM LCS Control Limits represent:
  - Multiple laboratories using their in-house procedures
  - Multiple versions of the methods
  - Multiple sample preparation/extraction/detection options

What about “Poor Performers”?

Well, what exactly is a “poor performer”?

Analytes for which recoveries were ≤ 15% (≥ 100%) or had RSD ≥ 25% were considered poor performers.

64 analytes were identified as poor performers. They were censored and do not appear in the QSM/QSAS Appendix C Tables.

Analyte	Method	Standard Deviation	Mean	Control Limits
Tungsten	6010 (Solid)	43	41	0-170
Zirconium	6010 (solid)	46	70	0-207
Propene	RSK-175 (water)	14	142	100-185

Some Additional Poor Performers

Analyte	Method	Standard Deviation	Mean	Control Limits
Merphos	8141 (water)	26	74	0-153
Monocrotophos	8141 (water)	35	55	0-160
Dinoseb	8151 (water)	37	52	0-162
Dalapon	8151 (water)	21	62	0-125
Benzaldehyde	8270 (solid)	30	56	0-146
Benzidine	8270 (solid)	20	38	0-100
Benzoic Acid	8270 (solid)	25	66	0-140
Phenol	8270 (water)	21	46	0-108

A Case for 1,4 Dioxane by Method 8260

Analyte	Method	Standard Deviation	Mean	Control Limits
1,4 Dioxane	8260 (solid)	14	96	55-138
1,4 Dioxane	8260 (water)	13	99	59-139
1,4 Dioxane	8270 (solid)	17	48	0-100
1,4 Dioxane	8270 (water)	14	47	4-90
1,4 Dioxane	8270 SIM (water)	16	57	10-105

Questions??