

DoD Laboratory Control Sample (LCS) Study Final Resolutions

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Purpose of Briefing



- ◆ Review the background of the Laboratory Control Sample (LCS) Study
- ◆ Summarize the policy implications for both DoD and our Laboratories



Background



- ◆ Under the leadership of the DoD Environmental Data Quality Workgroup (EDQW), data from 20 laboratories was collected starting in October 2000
- ◆ The effort focused on the LCS as a measure of the accuracy of analytical performance
- ◆ Data collection was coordinated by the American Council of Independent Laboratories (ACIL) to ensure confidentiality



Background



PURPOSE

- ◆ To develop LCS control limits that will serve as minimum acceptable limits for laboratories doing work for DoD
- ◆ To establish an objective benchmark for the development of alternative test methods and method modifications



Background

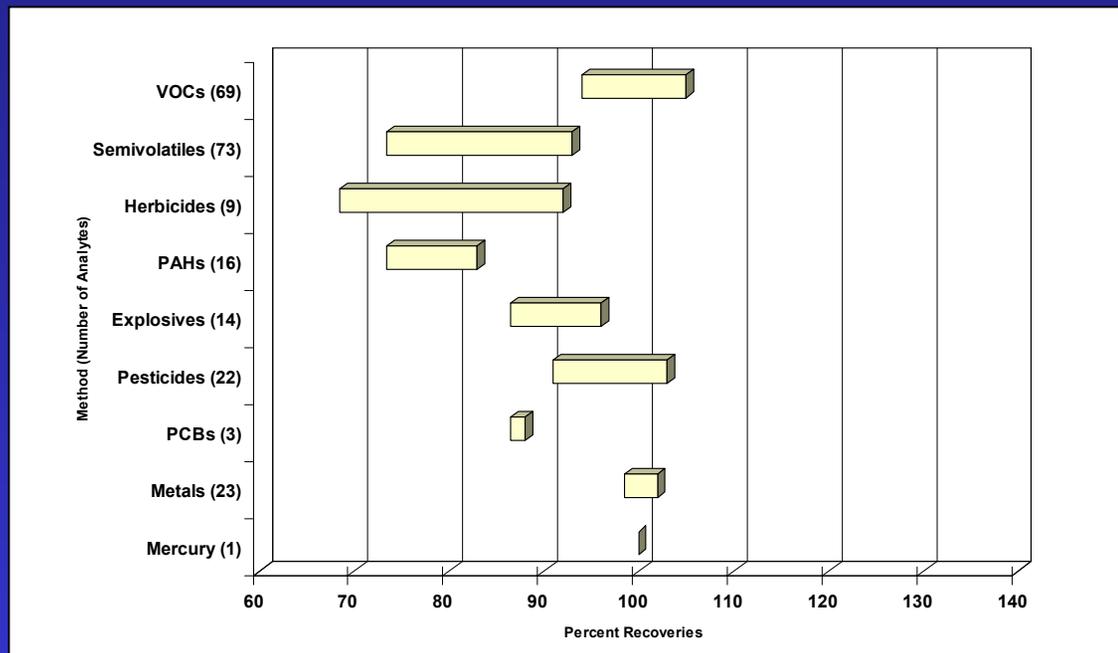


- ◆ The LCS Study focused on the nine most common USEPA SW-846 methods used in DoD work:
 - ❖ Semivolatiles 8270C
 - ❖ Volatiles 8260B
 - ❖ Herbicides 8151A
 - ❖ PAHs 8310
 - ❖ Explosives 8330
 - ❖ Pesticides 8081A
 - ❖ PCBs 8082
 - ❖ Metals 6010B
 - ❖ Mercury 7470A/71A



The Good News

- ◆ In general, Mean Recoveries were high, greater than 70% for the majority (93%) of the 454 total analytes





The Good News (cont'd)



- ◆ For Metals, the Interlaboratory control limits were narrower than method Quality Control requirements
 - ❖ Average Mean Recoveries were at or near 100%
- ◆ Overall, LCS control limits are more stringent than previously published results
 - ❖ The LCS study shows steady improvement in lab quality



A Less-Than-Perfect World



- ◆ Interlaboratory performance was inconsistent for most organic methods
- ◆ Analysis of Variance (ANOVA) results yielded high standard deviations, thus a greater degree of uncertainty
- ◆ The most problematic compound groups were the Herbicides & Semivolatiles



A Less-Than-Perfect World (cont'd)



- ◆ Poor Performers: calculated statistical lower control limits at or below 10%:
 - ❖ 4-Nitrophenol, Phenol (8270C Water)
 - ❖ 4-Chloroaniline, 3,3'Dichlorobenzidine (8270C Solid)
 - ❖ Tetryl (8330 Solid)
- ◆ However, calculated Failure Rates demonstrated that an LCS failure is more likely at the upper limit



Policy Decisions: Laboratories



- ◆ DoD requires that all analytes be spiked into the LCS
- ◆ 'Poor Performer' compounds will not cause a LCS failure, unless the compound is a project specific concern
- ◆ Metals control limits were expanded to include uncertainty in instrument calibration
- ◆ Control limits were set using 3 standard deviations around the mean, after outlier removal



Policy Decisions: Laboratories (cont'd)



To account for random error and long analyte lists, 5% of analytes outside of control limits is acceptable

- ❖ Must be random
- ❖ Must be within a four-sigma (Marginal Exceedance) limit
- ❖ Cannot be a project analyte of concern
- ❖ Surrogates: Marginal exceedances are not allowed



Policy Decisions: Laboratories (cont'd)



Allowable Number of Marginal Exceedances of LCS-Control Limits:

❖	> 90	5
❖	71 – 90	4
❖	51 – 70	3
❖	31 – 50	2
❖	11 – 30	1
❖	< 11	0



Policy Decisions: Laboratories (cont'd)



- ◆ Herbicides (method 8151A): Intralaboratory variability was the highest for all the methods studied; Non-parametric approach (5th and 95th percentiles) chosen to set control limits
- ◆ In-house control limits: Labs must still generate these as they are helpful for tracking performance
- ◆ In-house limits must meet or exceed DoD limits



Policy Implications: *What About DoD?*



- ◆ LCS Study reveals that extraction method is crucial to successful analysis, but is rarely discussed
- ◆ Long analyte lists produce a false sense of security
- ◆ If long analyte lists are desirable, then multiple extractions become a necessity
- ◆ Projects must focus only on analytes of concern and optimize for those analytes
- ◆ Laboratory - DoD client interaction paves the way for better data



A Final Caveat



- ◆ The LCS Study focuses on contract compliance/ batch acceptance.
- ◆ The study involved multiple laboratories and lacked a uniform population since SW-846 offers a number of choices in analyzing a particular analyte.
- ◆ The LCS study showed that SPE performed better than “salting out” as an extraction method for the analysis of explosives. This was the only case where one extraction method was shown to be preferable to another.



A Final Caveat



- ◆ Other extraction method choices (e.g. Soxhlet vs. Sonication) did not reveal any statistical difference due to limited data .
- ◆ “Confounding” of variables such as differing spike levels, cleanup methods, purge volumes, temperature programs, etc., could not be addressed.



In Conclusion



Version 2 of the DoD Quality Systems Manual (QSM) contains the LCS control limits as an appendix:

<https://www.denix.osd.mil/denix/denix.html>

A final copy of the LCS Study methodology is being prepared and will be released via DENIX in the near future.