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THE LEADER IN ENVIRONMENTAL TESTING

Making Progress on Detection, Quantitation and Calibration

Activities of the EMMEC

Richard Burrows

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EPA RFP

OVERVIEW INFORMATION

U.S. Environmental Protection Agency Office of the Science Advisor

Forum on Environmental Measurement

SUPPORT TO DEVELOP MEASUREMENT TOOLS, ACCREDITATION STANDARDS, AND TECHNICAL SUPPORT

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2.1 Develop Measurement Tools to Improve the Quality of Method Information, Understanding, and Flexibility

TNI proposes to:

Form an Environmental Measurement Methods Expert Committee chartered to develop consensus standards that will establish requirements for fundamental measurement practices such as Limit of Detection (LOD), Limit of Quantitation (LOQ), and instrument calibration to reduce quality system vulnerabilities.

Develop a Methods Interpretation Request process, comparable to the process already used by TNI for responding to interpretation requests on the TNI Standard.

Build a Methods Compendium that would contain, or link to, all test methods used for environmental analyses.

Work with EPA's Environmental Laboratory Advisory Board.

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EMMEC

Environmental Measurement Methods Expert Committee

A TNI subcommittee

Specific request in EPA RFP to develop tools for detection, quantitation and calibration

EMMEC Charter

The objectives are to:

create and adopt standards to support a strong technical approach to quantitation, detection and calibration;

develop standards that are useable across various EPA and state programs.

EMMEC Members

Richard Burrows, Chair
TestAmerica

Ken Jackson, Program Administrator
The NELAC Institute

Brooke Connor
US Geological Survey

Dan Dickinson
New York State DOH

Tim Fitzpatrick
Florida DEP

Nancy Grams
Advanced Earth Technologies, Inc.

Anand Mudambi
US EPA

John Phillips
Ford Motor Co

Lee Wolf
Columbia Analytical Services

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Calibration

Quantitation

Detection

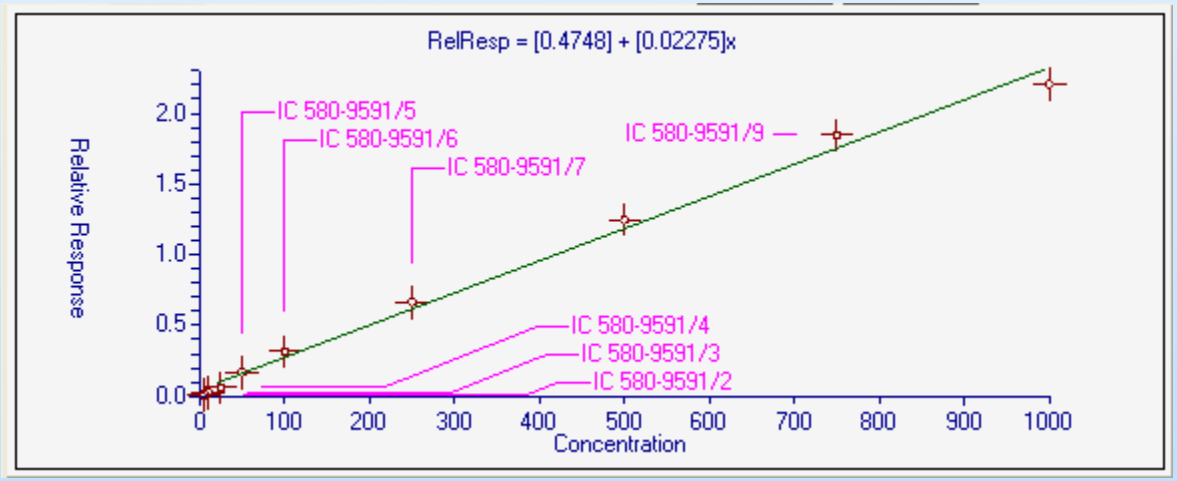
Detection

Calibration

Lots of current activity

Less controversial (?)

Change of pace!



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Weaknesses of calibration practices in EPA methods

Some older methods have very little guidance

Inject three standards, prepare a calibration curve

Some methods allow the use of a number of standards that would generally be considered inadequate

Evaluation of a calibration curve is almost always done with the correlation coefficient or coefficient of determination

There is little if any guidance on which type of calibration curve to use

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Weaknesses of calibration practices in EPA methods

There is often little verification that results at the high or low ends of the calibration curve are accurate

Unweighted linear regression is allowed and even preferred

Methods often do not have controls over deletion of points from a curve

There is no distinction made between calibration requirements for detected and not-detected analytes

Only single replicates are required for each level

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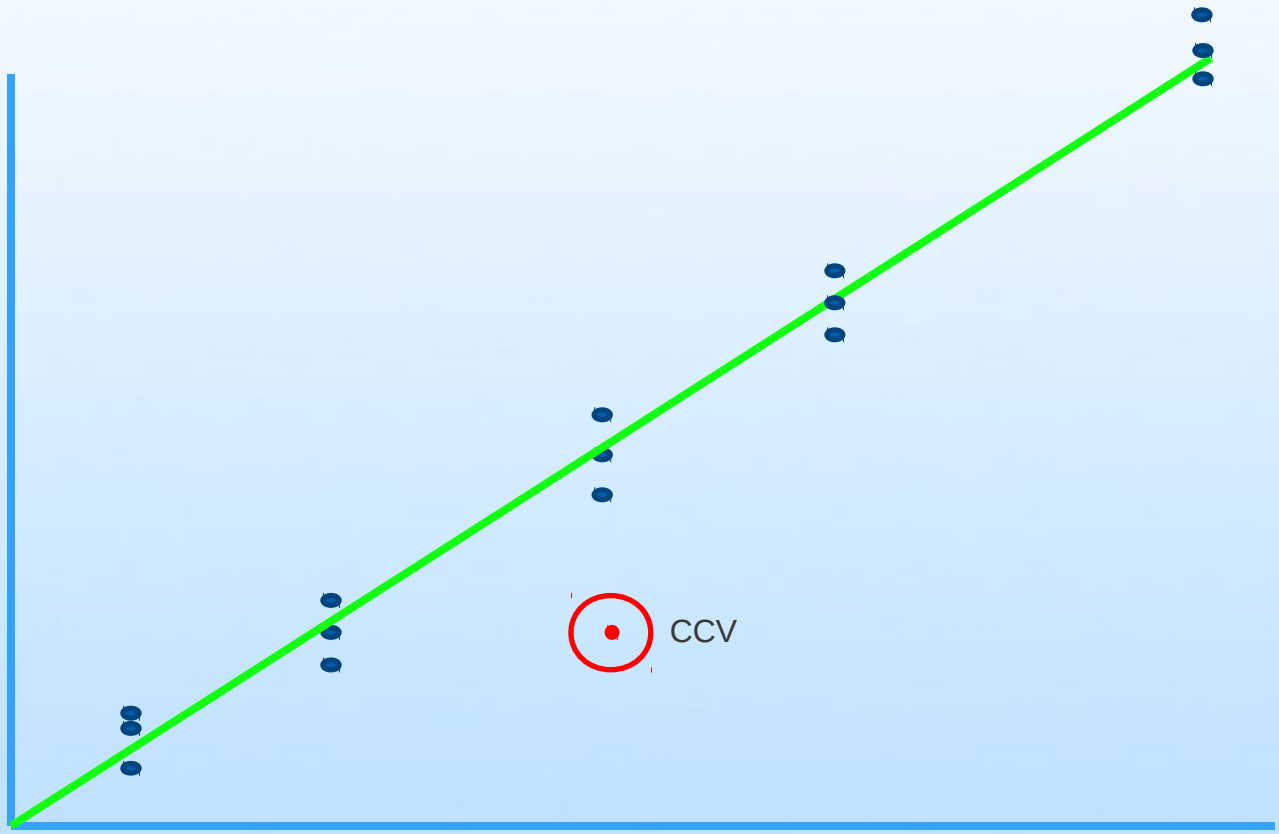
Multi replicate calibration



Issues with Calibration

Analyze at least 5 points

RSD, linear regression, quadratic regression

r, r2 > 0.990 (0.995)

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The curve that cannot fail

Conc	Resp
1	0.00
2	0.00
3	0.00
4	0.00
5	0.00
10	0.00
100	117
slope	0.81564
corr	0.99679
int	4.16667

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GC PCB				
	Ave	Lin	Lin1/X	Lin 1/X2
50	61%	243%	45%	4%
250	11%	9%	21%	17%
500	1%	12%	22%	13%
1000	15%	10%	10%	0%
2000	25%	4%	0%	10%
3000	32%	3%	8%	17%
	34%	122%	28%	15%
	0.79	0.994	0.987	0.98

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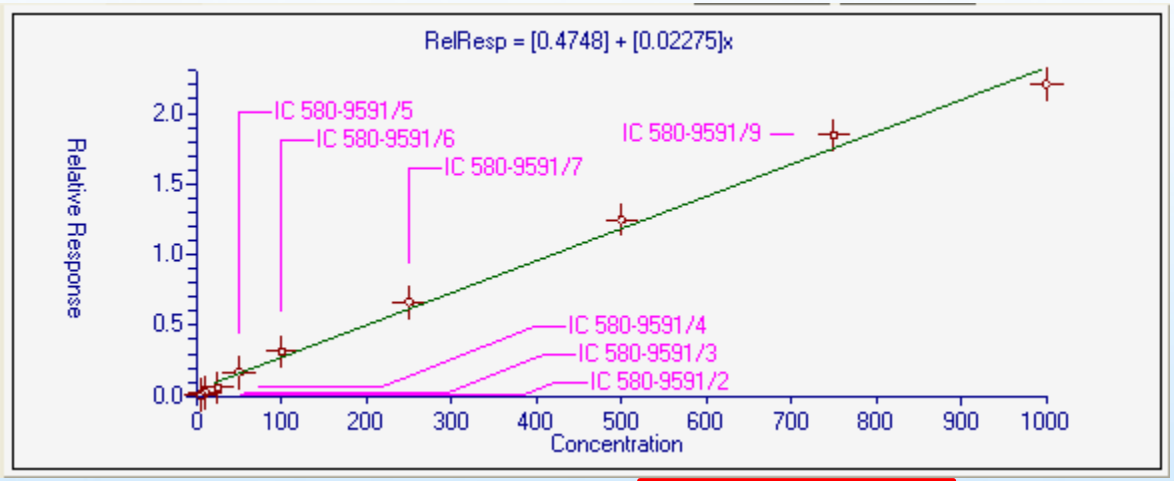
Calibration issues



r= 0.997, r2 = 0.994

RSE = 179%

Calibration Standard Levels

Level	Used	Amount	Area	ISArea	%Error
IC 580-9591/2	☑	5	1348	618332	421.63
IC 580-9591/3	☑	10	3250	647316	198.43
IC 580-9591/4	☑	25	7697	646400	78.87
IC 580-9591/5	☑	50	23729	700099	7.13
IC 580-9591/6	☑	100	47131	748204	17.47
IC 580-9591/7	☑	250	111297	833662	8.93
IC 580-9591/8	☑	500	229185	917698	5.52
IC 580-9591/9	☑	750	371628	1005615	5.43
IC 580-9591/10	☑	1000	499631	1131444	5.11

421.63

198.43

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Solutions to Calibration

Calculate “readback” for each level

Recent drinking water methods

Recent SW-846 methods

Pros

Provides an indication of the error introduced at each level

Conceptually straightforward

Cons

Lots of numbers!

Difficult to compare different curve types

Need to be careful with criteria

Solutions to calibration

RSE

Extends applicability of RSD (used for average curve) to all other curve types

Pros

Allows easy comparison of curve types

Will indicate failing calibration if any point (high or low concentration) has a high deviation from the curve

Can use same criteria as RSD

Cons

Not currently available in most chromatographic data systems

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Challenges

Tools that we would like to use for calibration assessment are not currently available in most instrument software

Need to be compliant with EPA analytical methods

Need to be consistent with Quality Systems standards

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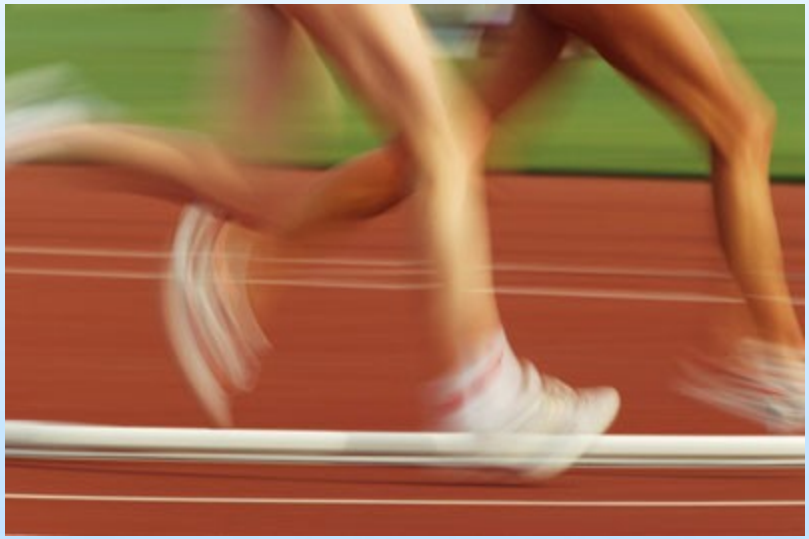
Goal

Working draft standard for consideration at Sarasota TNI meeting, January 2012



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Questions?