What we need a procedure to do?

i. Does the procedure provide an explicit estimate of bias at L_Q for limits that must be verifiable by labs at those limits?

ii. Does the procedure provide an explicit estimate of precision at L_Q for limits that must be verifiable by labs at those limits?

iii. Does the procedure provide an explicit false positive rate for L_C?

iv. Does the procedure provide an explicit false negative rate at L_C for the true value at L_D or L_Q that must be observed in labs at L_C for the estimated values of L_D or L_Q?

v. Does the procedure provide that qualitative identification criteria defined in the analytical method are met at the determined detection and quantitation limits?

vi. Does the procedure adequately represent routine variability in lab performance?

vii. Does the procedure perform on-going verification of estimates?

viii. Is the procedure capable of calculating limits using matrices other than lab reagent grade water?

ix. Does the procedure use only data that results from test methods conducted in their entirety?

x. Does the procedure explicitly adjust or account for situations where method blanks always return a non-zero result/response?

xi. Does the procedure explicitly adjust or account for situations where method blanks are intermittently contaminated?

xii. Is the procedure clearly written with enough detail so that most users can understand and implement them?

xiii. Is the procedure cost effective?

xiv. Does the procedure assess multi-laboratory and inter-laboratory variability when data from more than one lab is used?

xv. Is the procedure applicable to all users and test methods?

What we need a procedure to do!

1. The procedure must provide an explicit estimate of bias at L_Q for limits that must be verifiable by labs at those limits.

2. The procedure must provide an explicit estimate of precision at L_Q for limits that must be verifiable by labs at those limits.

3. The procedure must provide an explicit false positive rate for L_C.

4. The procedure must provide an explicit false negative rate at L_C for the true value at L_D or L_Q that must be observed in labs at L_C for the estimated values of L_D or L_Q.

5. The procedure must provide that qualitative identification criteria defined in the analytical method are met at the determined detection and quantitation limits.

6. The procedure must adequately represent routine variability in lab performance.

7. The procedure must perform on-going verification of estimates.

8. The procedure must be capable of calculating limits using matrices other than lab reagent grade water.

9. The procedure must use only data that results from test methods conducted in their entirety.

10. The procedure must explicitly adjust or account for situations where method blanks always return a non-zero result/response.

11. The procedure must explicitly adjust or account for situations where method blanks are intermittently contaminated.

12. The procedure must be clearly written with enough detail so that most users can understand and implement them.

13. The procedure does not add a significant additional burden to the laboratory.

14. The procedure must assess multi-laboratory and inter-laboratory variability when data from more than one lab is used.

15. The procedure must be applicable to all users and test methods.

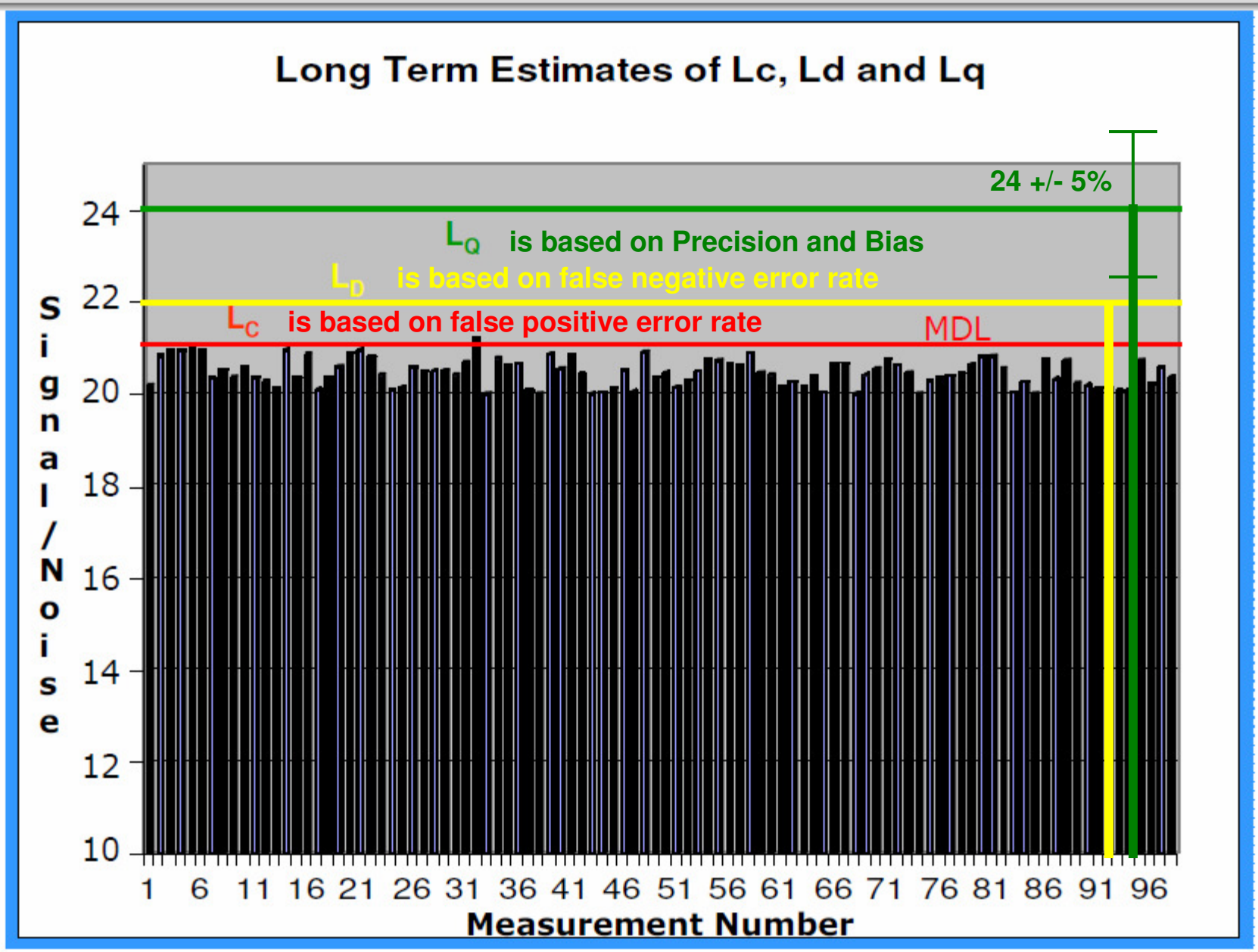


FIGURE 2

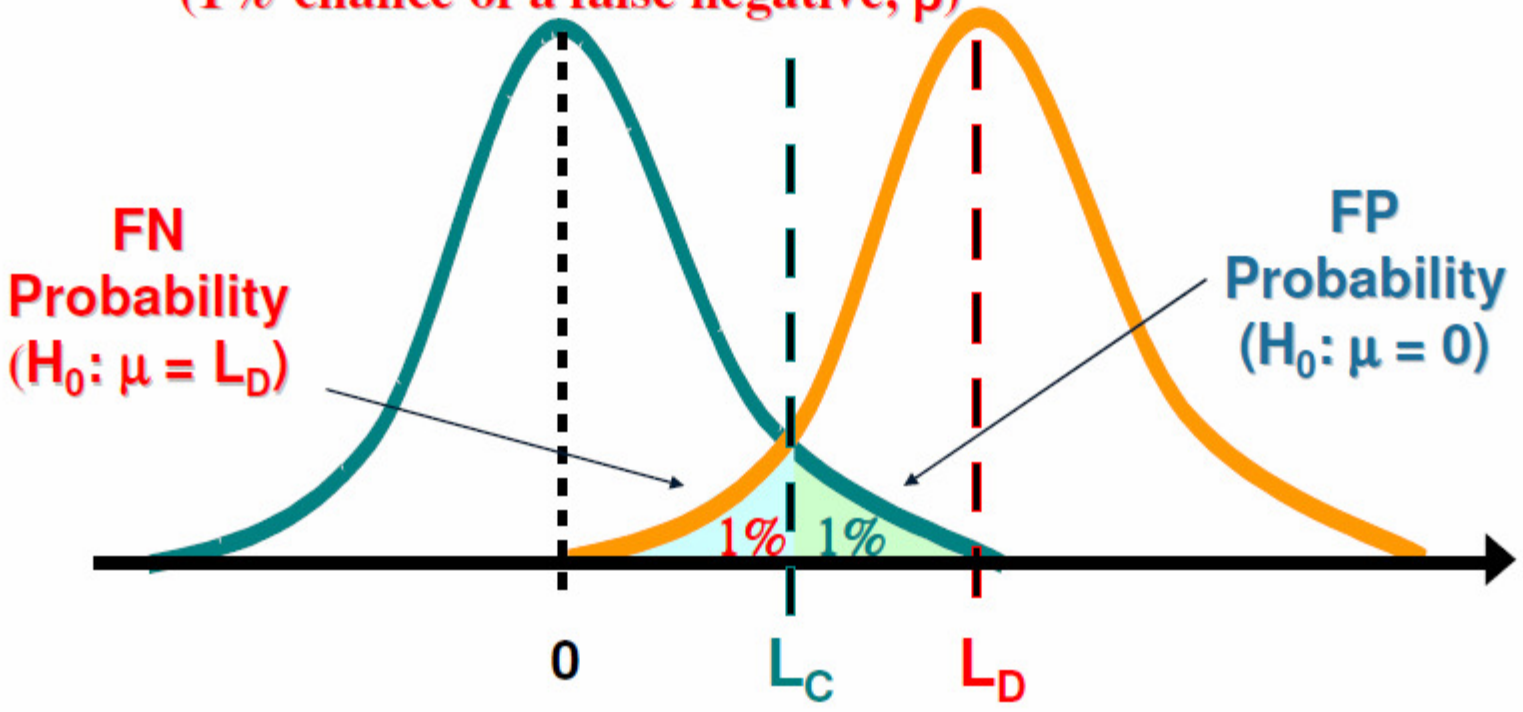
Conceptualization of L_C and L_D

L_C = Smallest value > 0 with 99% confidence

(1% chance of a false positive, α)

L_D = Smallest value to give results > L_C with 99% confidence

(1% chance of a false negative, β)



What we need a procedure to do!

1. **The procedure must provide an explicit estimate of bias at L_Q for limits that must be verifiable by labs at those limits.**

   Example: *90% to 110% Recovery*

2. **The procedure must provide an explicit estimate of precision at L_Q for limits that must be verifiable by labs at those limits.**

   Example: *10% RSD*

3. **The procedure must provide an explicit false positive rate for L_C.**

   Example: *1% FP error rate*

4. **The procedure must provide an explicit false negative rate at L_C for the true value at L_D or L_Q that must be observed in labs at L_C for the estimated values of L_D or L_Q.** ***Example:*** *5% FN error rate*

5. **The procedure must provide that qualitative identification criteria defined in the analytical method are met at the determined detection and quantitation limits.** ***Example:*** *m/Z and ion ratios for GC/MS*

What we need a procedure to do!

6. The procedure must adequately represent routine variability in lab performance. ***Example:*** *List of within lab variability*

14. The procedure must assess multi-laboratory and inter-laboratory variability when data from more than one lab is used.

Example: *List of within mulit-laboratory variability*

Example: *Difference between inter & intra-laboratory variability*

7. The procedure must perform on-going verification of estimates.

Example: *Time (monthly/annually) or number of analyses or both*

8. The procedure must be capable of calculating limits using matrices other than lab reagent grade water.

Example: *Groundwater, Wastewater, Soil, Oil*

9. The procedure must use only data that results from test methods conducted in their entirety.

Example: *Must include extraction, clean-up and analysis*

What we need a procedure to do!

10. The procedure must explicitly adjust or account for situations where method blanks always return a non-zero result/response.

Example: *Atomic Absorption or ICP/AES*

11. The procedure must explicitly adjust or account for situations where method blanks are intermittently contaminated.

Example: *Mercury by 1631, Dioxin & Furans by 1613 or PCBs by 1668*

12. The procedure must be clearly written with enough detail so that most users can understand and implement them.

Somewhat subjective, but can be evaluated

13. The procedure does not add a significant additional burden to the laboratory. *Somewhat subjective, but can be evaluated*

Also: *Does the procedure require additional data or information to be collected by the laboratory and does this data support other objectives of the laboratory such as estimating method uncertainty?*

15. The procedure must be applicable to all users and test methods.

This may or may not be possible!

Measuring Variability in an individual laboratory

- Must have replicate analyses of same sample or standard.
- Collected over enough time to capture variability in performance.
- Incorporate variability due to the use of multiple instruments.
- Incorporate variability due to use of multiple analysts.
- Adjust or account for recovery.
- Consistency or uniformity in the choices for outlier tests.

Measuring Variability in across laboratories

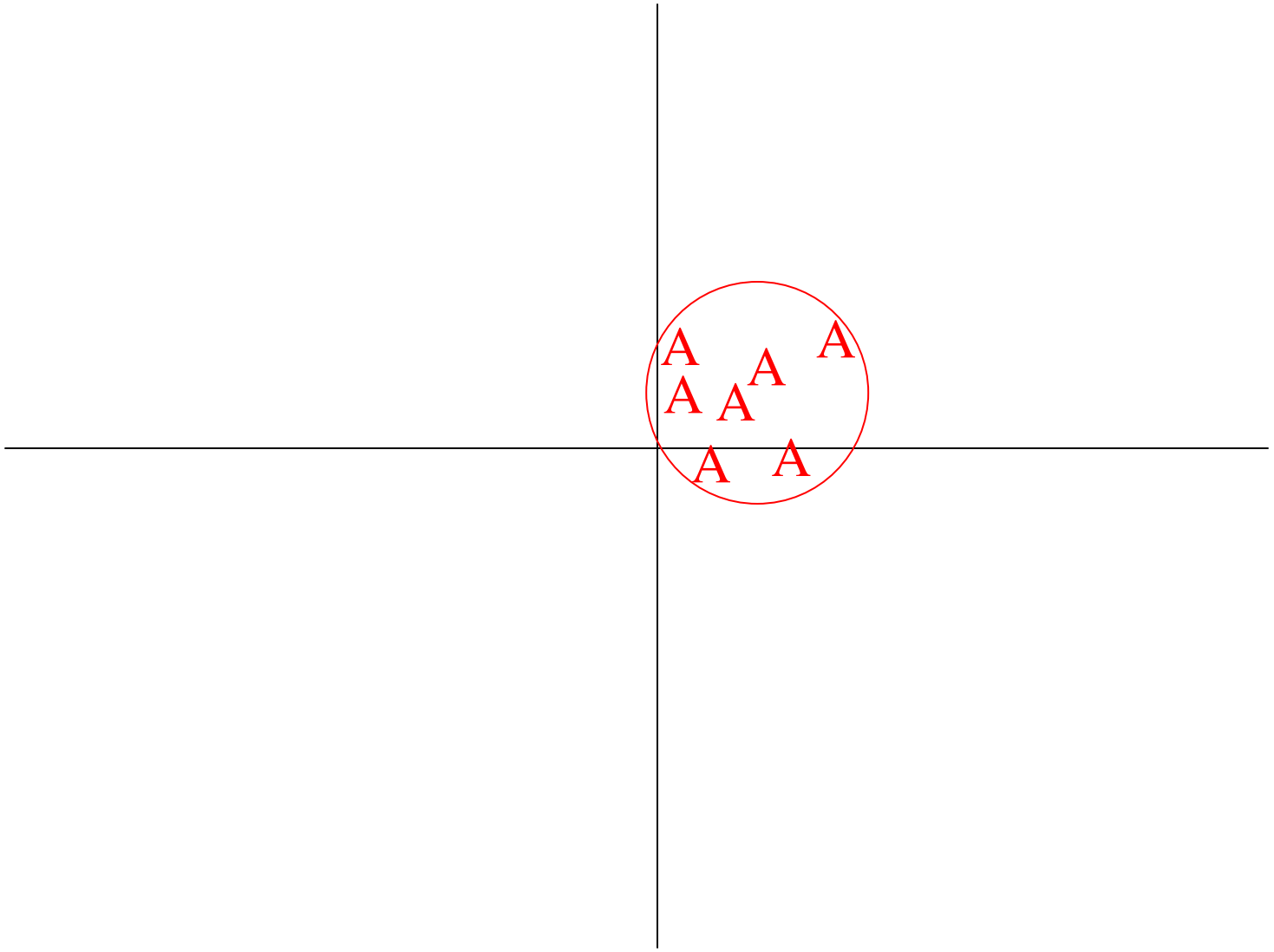
• Incorporate variability occurring across laboratories (include all sources of variability listed for individual laboratories).

• Includes slight differences in test method procedures.

• Address the number of different concentrations (spikes) that are used between laboratories.

• Address the spacing of concentrations (spikes) that are used between laboratories.

• Address varying numbers of replicates per concentration (spike) that are used between laboratories.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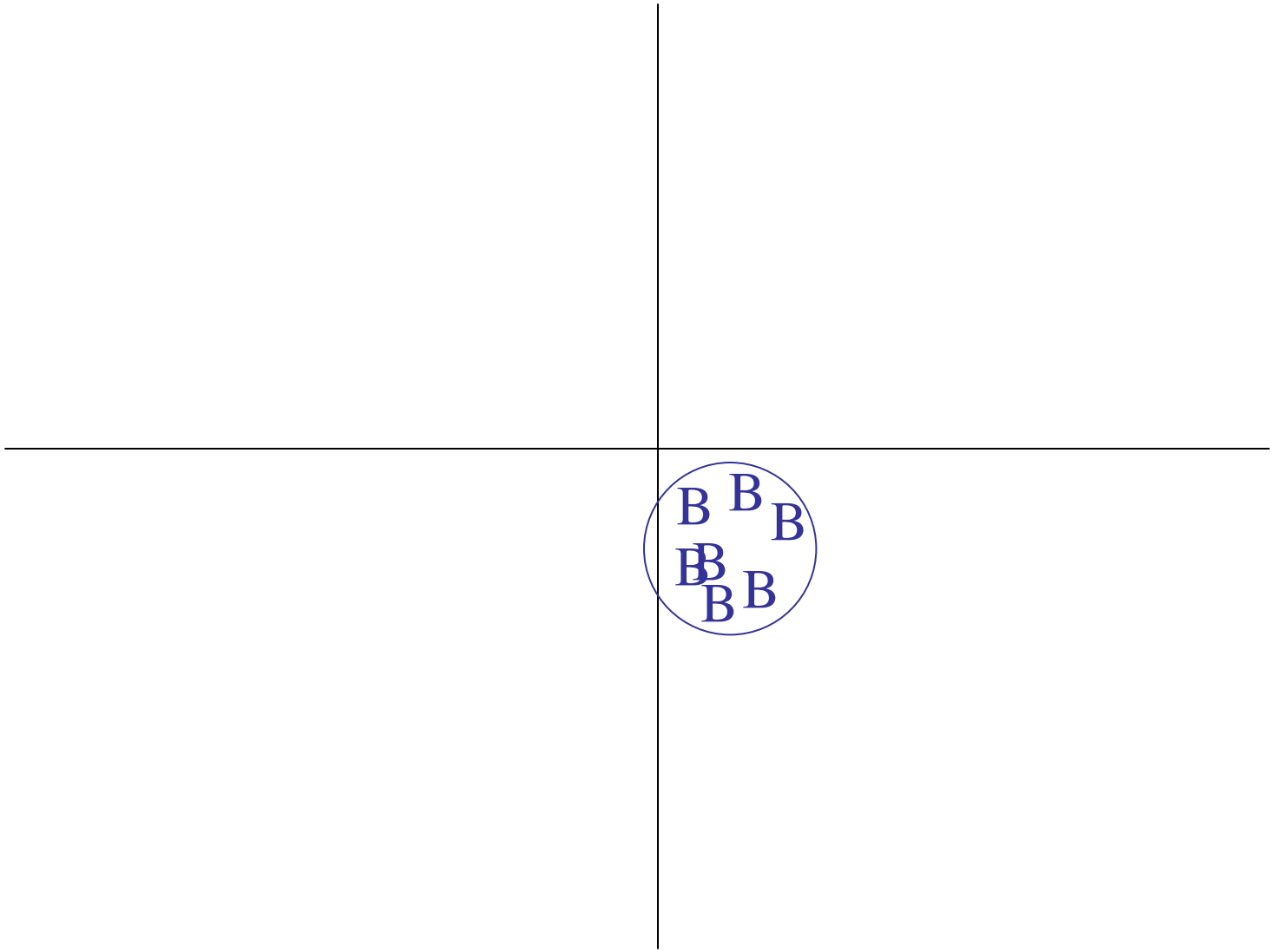
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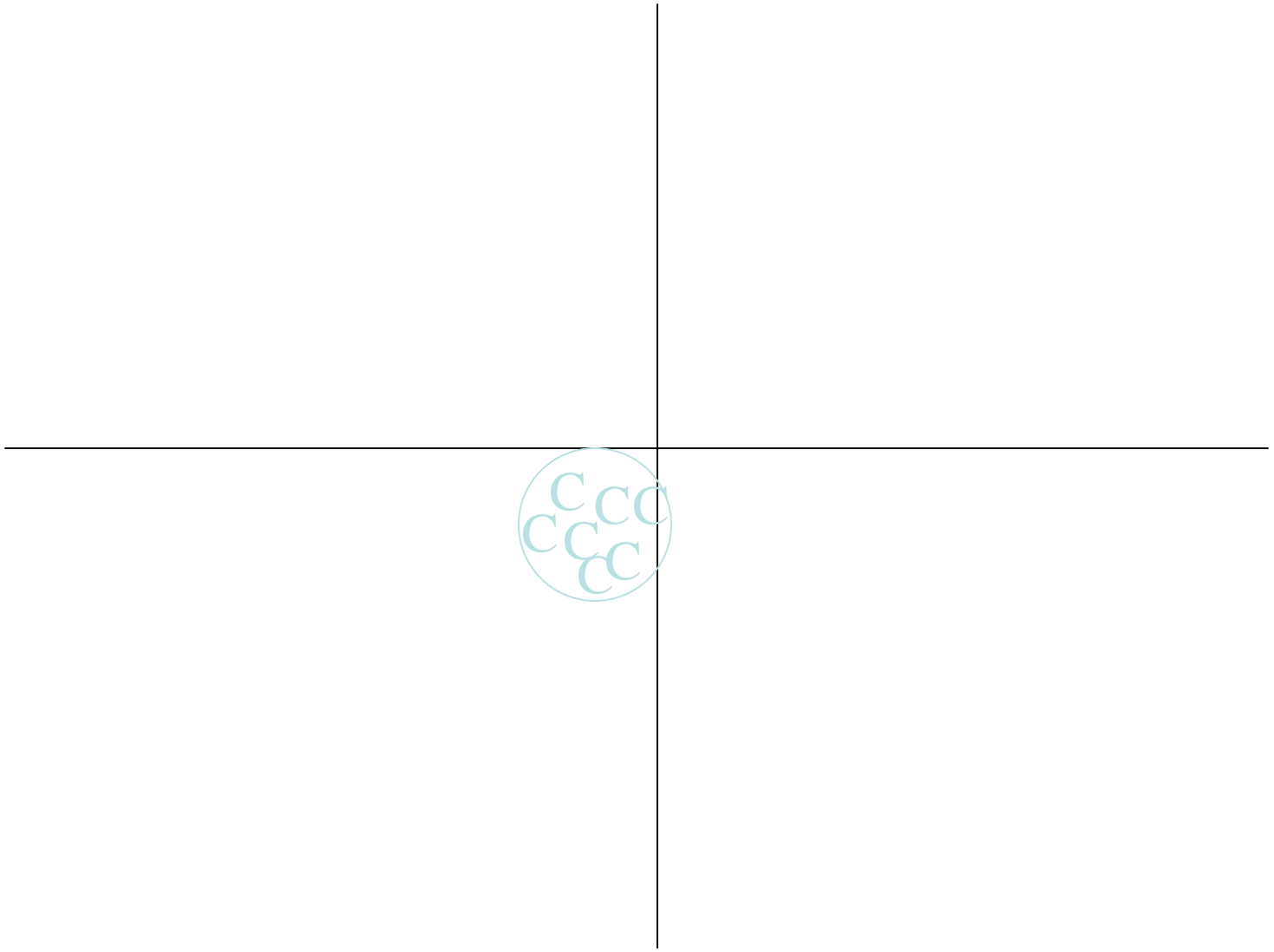

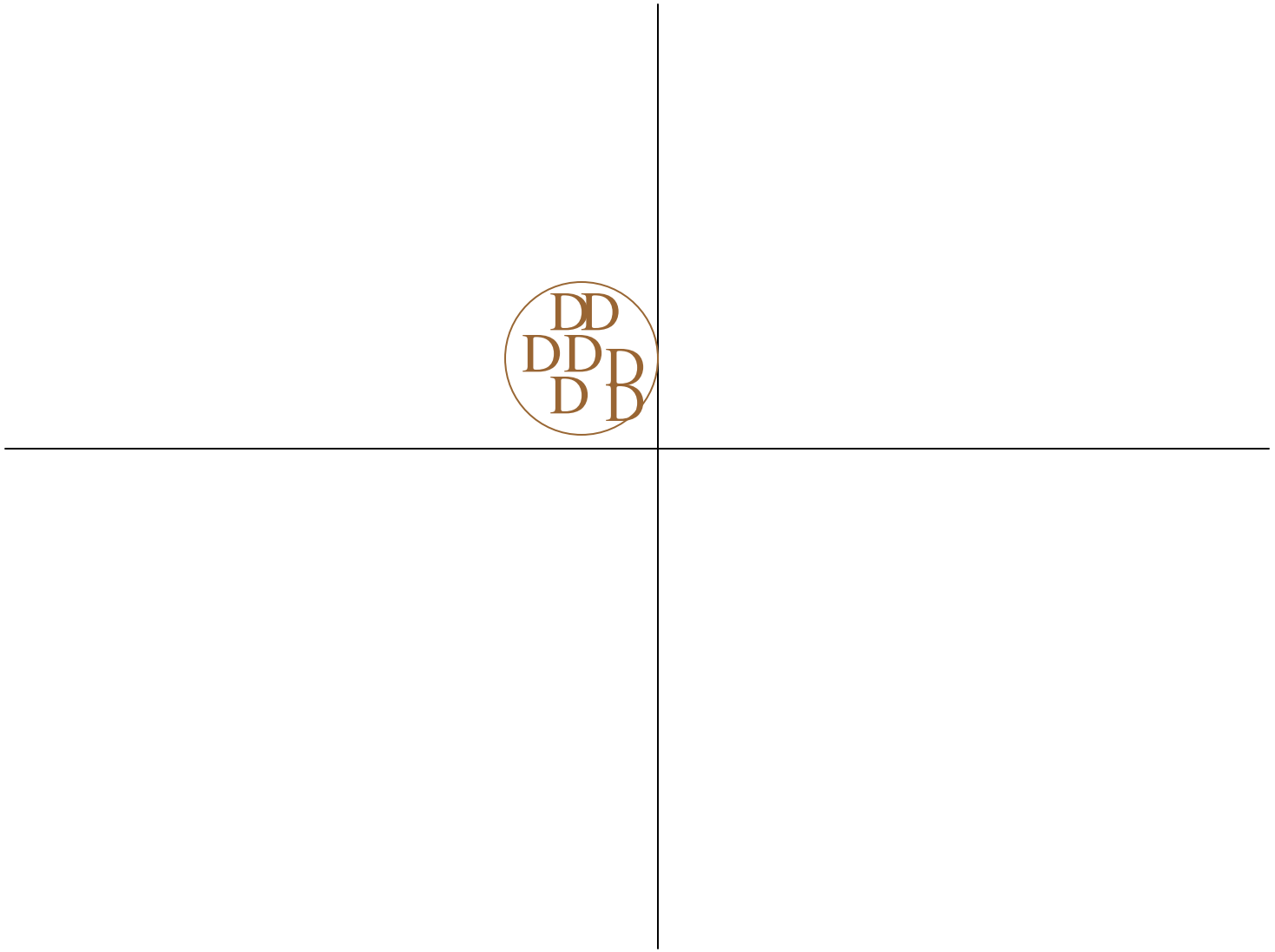

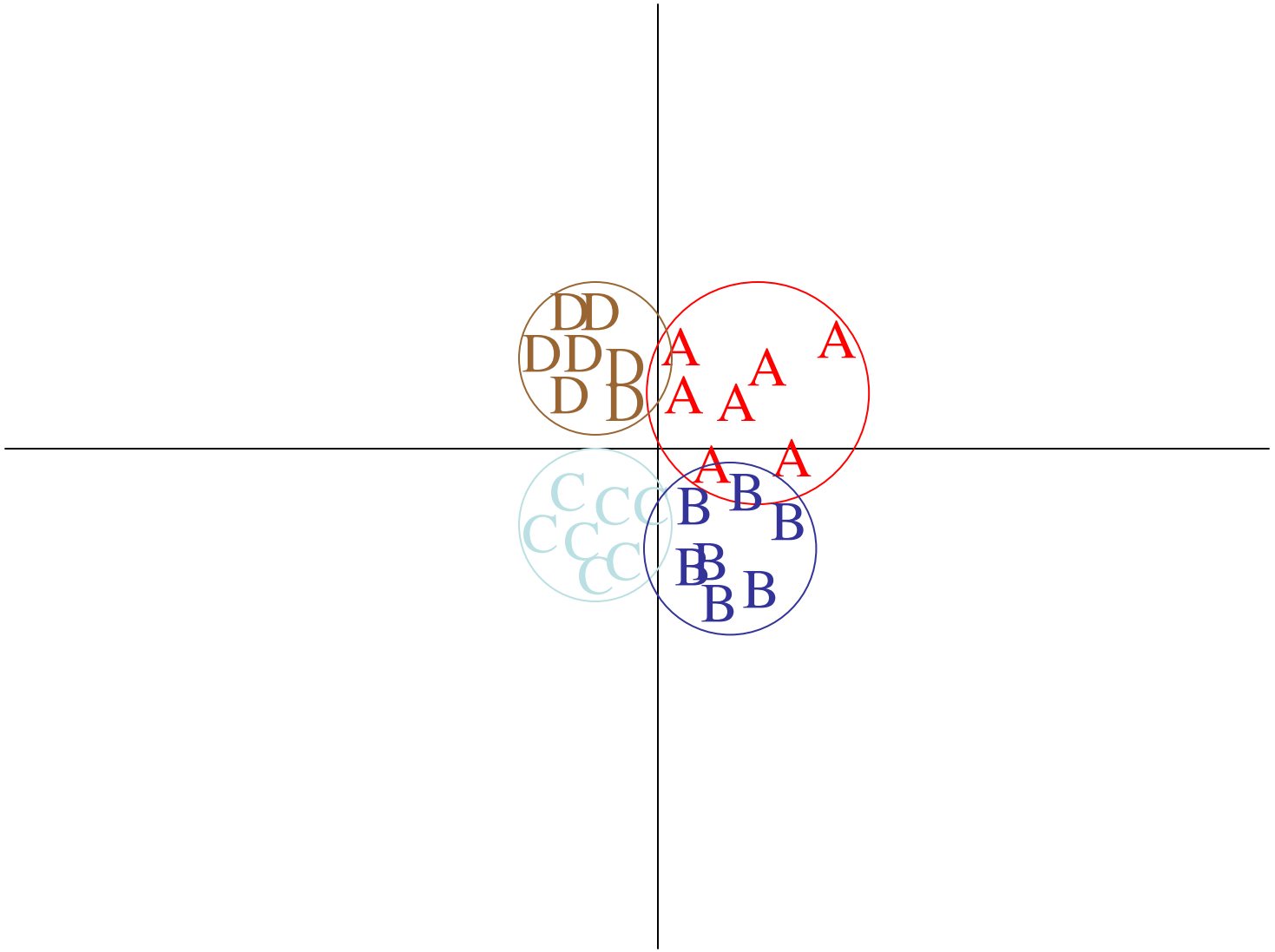
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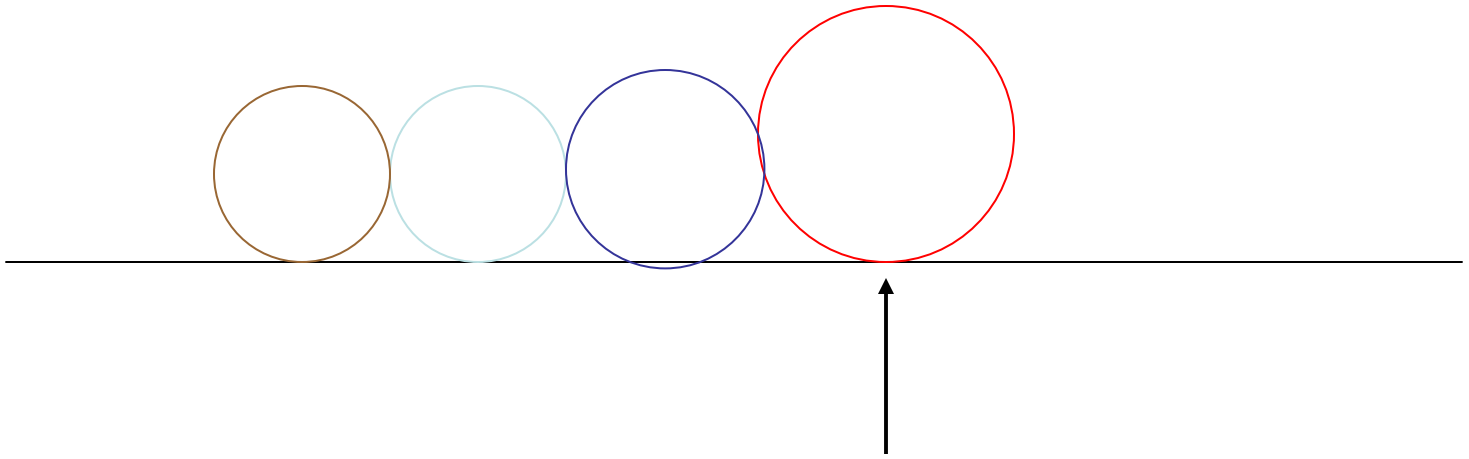
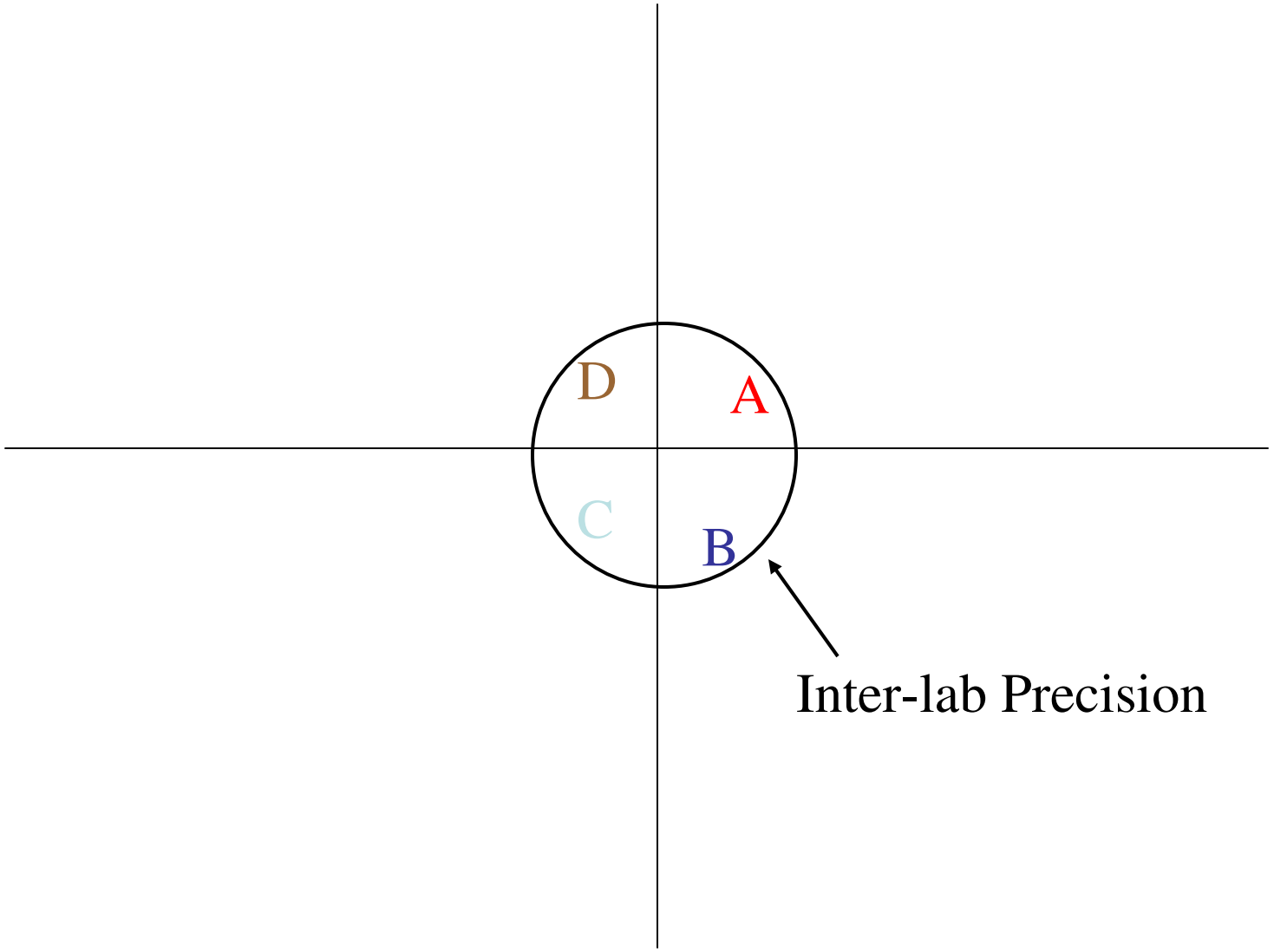


Precision from a family of
Qualified laboratories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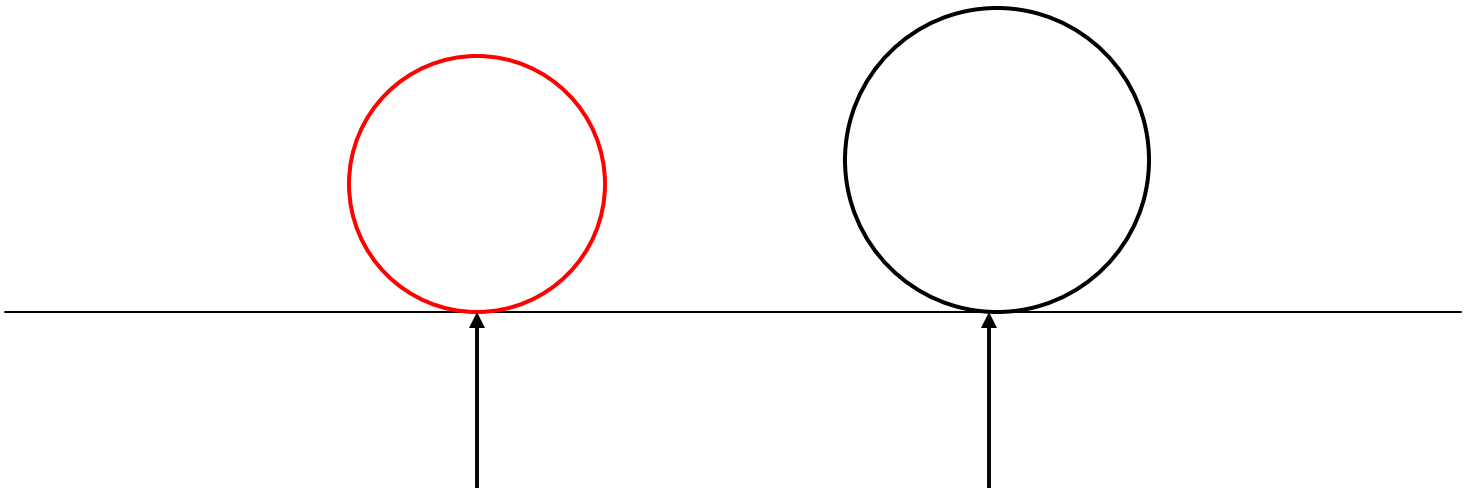
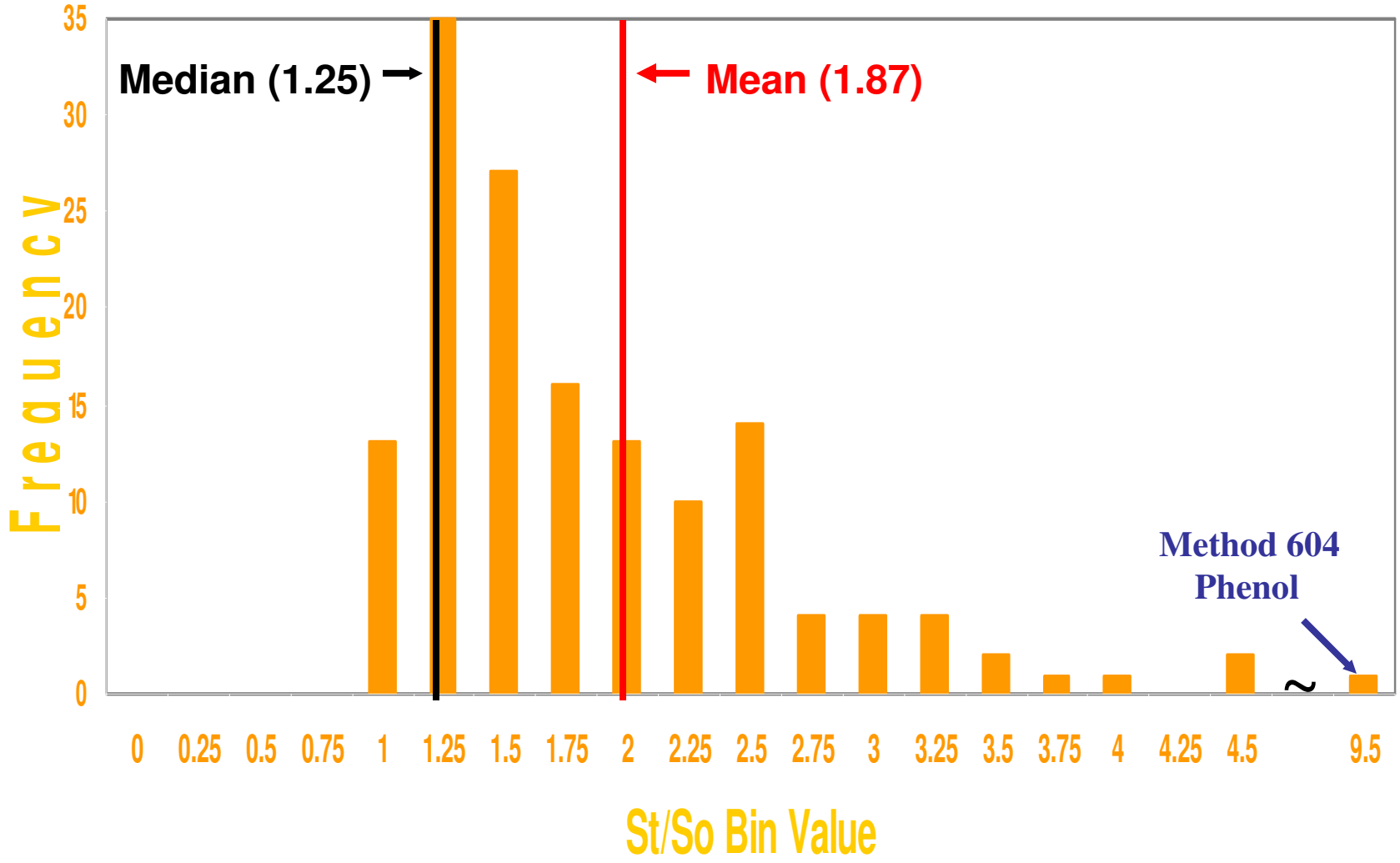
Inter-lab Precision



Precision from a family of qualified laboratories

True Inter-lab Precision

Interlab/Intralab Standard Deviation (EPRI, EPA and AWWA Data Sets)



1. Provide an explicit estimate of bias at LQ for limits that must be verifiable by labs at those limits.

To be evaluated by:

Reviewing procedure(s) and specifically identifying the quantitative limit for bias at LQ that is tested in the pilot study.

Requiring labs to analyze samples (spikes, blind or otherwise as appropriate) and comparing observed bias to that cited by the procedure(s).

2. Provide an explicit estimate of precision at LQ for limits that must be verifiable by labs at those limits.

To be evaluated by:

Reviewing procedure(s) and specifically identifying the quantitative limit for precision at LQ that is tested in the pilot study.

Requiring labs to analyze samples (spikes, blind or otherwise as appropriate) and comparing observed precision to that cited by the procedure(s).

See Appendix for specific MQOs adopted by the committee for the pilot study

6. Adequately represent routine variability in lab performance.

To be evaluated by determining whether the procedures:

Use data to calculate limits that are collected over enough time to capture variability in performance relative to MQOs.
Recalculate limits at a frequency that captures variability in performance relative to MQOs.
Incorporate variability due to the use of multiple instruments per lab.
Incorporate variability due to use of multiple analysts per lab.
Incorporate variability occurring across laboratories (not for single lab. procedure).
Adjust or account for recovery.
Provide recommendations or limit choices for outlier tests.
Address varying numbers of different concentrations (spikes) that can be Used between laboratories (may only apply to multi/inter lab procedures).
Address varying numbers of replicates per concentration (spike) that can be used between laboratories (may only apply to multi/inter lab procedures).
Address varying combinations of concentrations (spikes) that can be used between laboratories (may only apply to multi/inter lab procedures).

Mission Statement:

Develop useful and easily implemented measurement tools that will improve the quality of data generated by environmental testing laboratories.

Create and adopt standards to support a strong technical approach to quantitation.

Create and adopt standards to support a strong technical approach to detection.

Create and adopt standards to support a strong technical approach to calibration.

Any standard developed should incorporate data quality objectives.

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

The Steering Committee agreed on the following general objective:

"Our approach will be to improve the technical quality of methodologies that are currently in use, in a way that minimizes the impact on laboratories while maximizing improvements in the quantitative estimates (LOD, LOQ, and calibration."

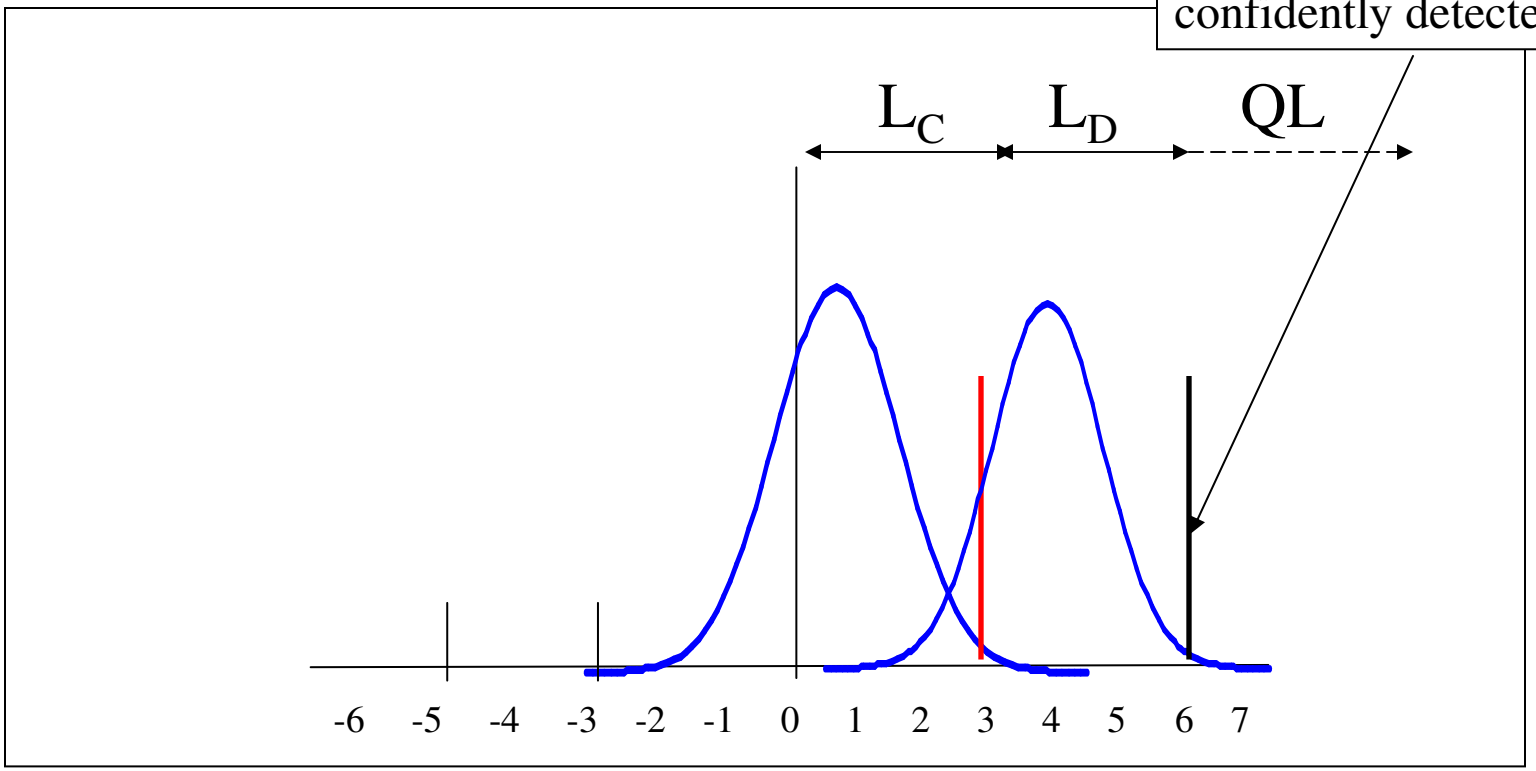
OR (Proposed)

"To improve the technical quality of environmental testing methodologies by providing tools (e.g., detection, quantitation and calibration) that assure the quality of data, which may be adopted by federal and state regulatory agencies. It is important that a balance between impact on laboratories and improvement in technical quality be maintained during this process."

		Single-lab Procedures				Inter/Multi-lab Procedures	
	Characteristics	ACIL	MDL/ML	CONSENSUS GROUP	LAB Q/C	LCMRL/H-V	IDE/IQE
1	provide an explicit estimate of bias at L_Q for limits that must be verifiable by labs at those limits.	Y	Y	Y	Y	Y	Y
2	provide an explicit estimate of precision at L_Q for limits that must be verifiable by labs at those limits.	Y	Y	Y	Y	Y	Y
3	provide an explicit false positive rate for L_C.	Y	Y/N	Y	Y	N/Y	Y/N
4	provide an explicit false negative rate at L_C for the true value at L_D or L_Q that must be observed in labs at L_C for the estimated values of L_D or L_Q.	Y	N	Y	Y	N/Y	Y/N
5	provide that qualitative identification criteria defined in the analytical method are met at the determined detection and quantitation limits.	Y	N	Y	N	Y/N	Y
6	adequately represent routine variability in lab performance.	Y	MAYBE	Y	Y	MAYBE	Y
7	perform on-going verification of estimates.	Y	Y	Y	Y	Y	Y
8	be capable of calculating limits using matrices other than lab reagent grade water.	Y	Y	Y	Y	Y	Y
9	use only data that results from test methods conducted in their entirety.	Y	Y	Y	Y	Y	Y
10	explicitly adjust or account for situations where method blanks always return a non-zero result/response.	Y	N	Y	MAYBE	N	Y
11	explicitly adjust or account for situations where method blanks are intermittently contaminated.	MAYBE	N	MAYBE	N	N	N
12	be clearly written with enough detail so that most users can understand and implement them.	Y	Y	Y	Y	Y	Y
13	be cost effective.	Y	Y	Y	Y	Y	MAYBE
14	assess multi/inter-laboratory variability when data from more than one lab is used.	MAYBE	MAYBE	MAYBE	N	Y	MAYBE
15	be applicable to all users and test methods.	Y	Y	Y	Y	Y	Y

QL and L_D

Concentration at which we are fairly sure that the result can be confidently detected



$QL >= L_D = 2 \times L_C$

QL must be greater than or equal to L_D where precision and bias MQOs are achieved.