

Reporting Limit (RL)

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Purpose

- **What is Reporting Limit (RL)?**
- **Why is RL needed?**
- **How is RL established?**
- **Applications and case study**
- **Major issues**
- **Summary**



Introduction

- **Reporting Limit (RL) is widely used but not clearly defined. For example:**
 - **RL – the minimum value below which data are documented as non-detects** – EPA OW, Office of Resource Conservation and Recovery (ORCR), and Office of Enforcement and Compliance Assurance (OECA)
 - **RL – the minimum value of the calibration range. Analyte detections between the detection limit and the reporting limit are reported as estimated** – EPA ORD and ORCR
 - **RL > MDL and the client and/or data-users determine RL**
– California Department of Health
- **Many projects improperly set RL at contract lab's LOQ**



RL in DoD QSM

- **DoD QSM Version 4.2:**
 - **RL – a client-specified lowest concentration value that meets project requirements for quantitative data with known precision and bias for a specific analyte in a specific matrix**
 - **RL = Client-specified quantitation limit that meets project data quality needs**
 - **RL is clearly defined in the DoD QSM but**
..... without determination procedure



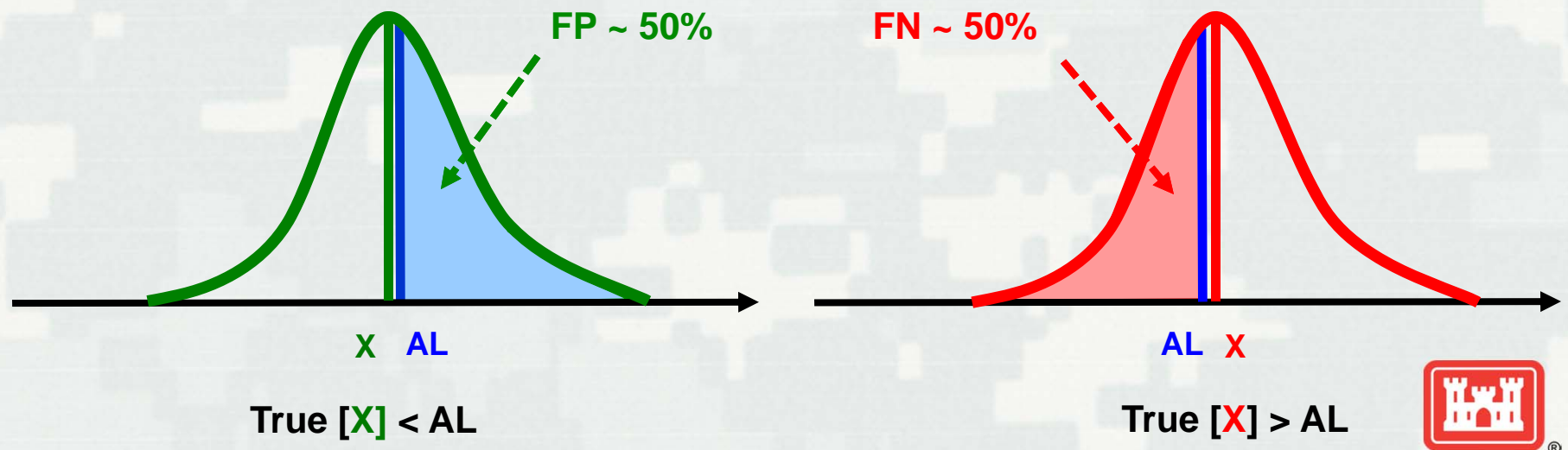
Project-Specific RL

- Why is a Project-Specific RL needed?
 - **To reliably determine if a site is CLEAN**
 - ✓ CLEAN: $[X] < \text{Regulatory Level, Background Level, Risk-Based Level, etc., i.e., Action Level (AL)}$
 - ✓ Procedure: comparison of $[X]$, mean, etc. with AL
 - ✓ Need to determine or estimate true analyte concentration with uncertainty at Project-Specific RL (i.e., Decision Level)
 - **To select analytical methods and labs**



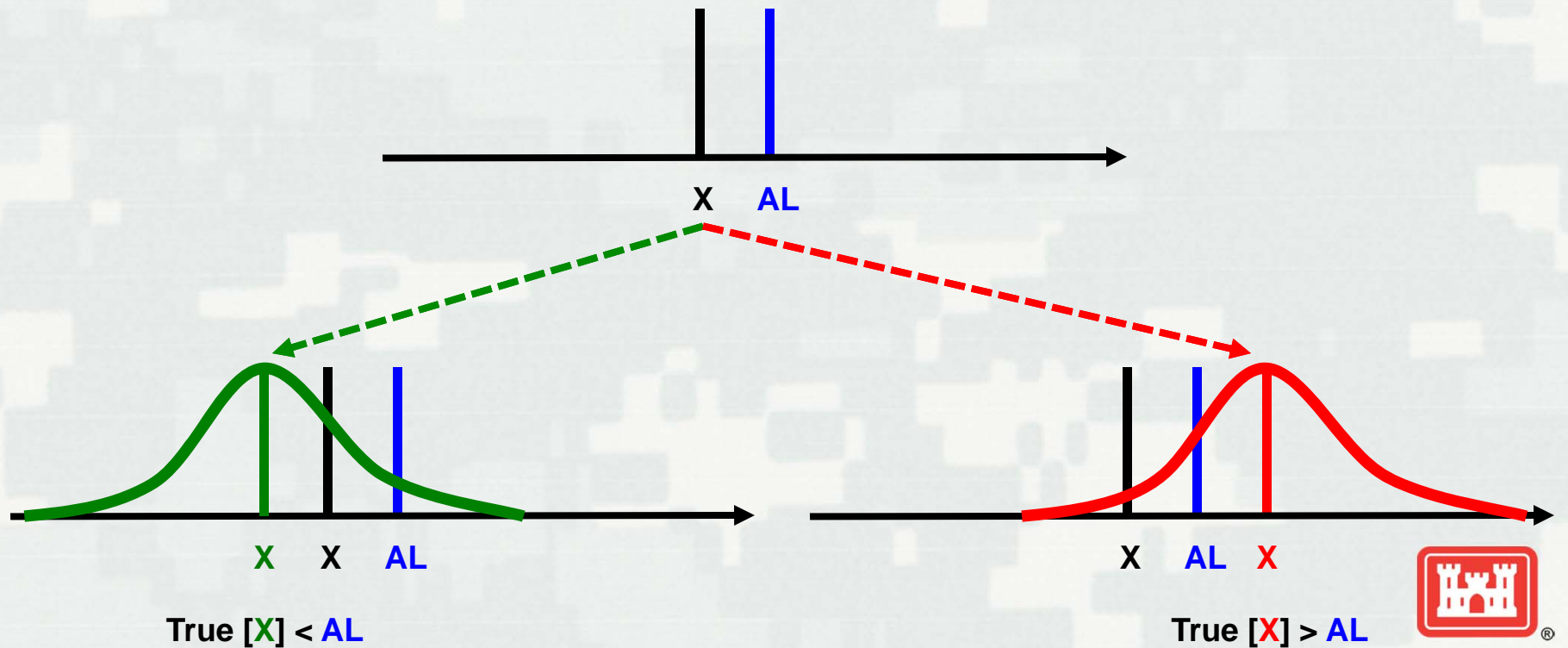
May RL = AL?

- To reliably determine if a site is clean, one needs to determine if $[X] < AL$
- Problems with $RL = AL$



Is $[X] < AL$?

- Is $[X] < AL$? $[X]$ = single/multi-measurements

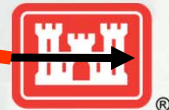
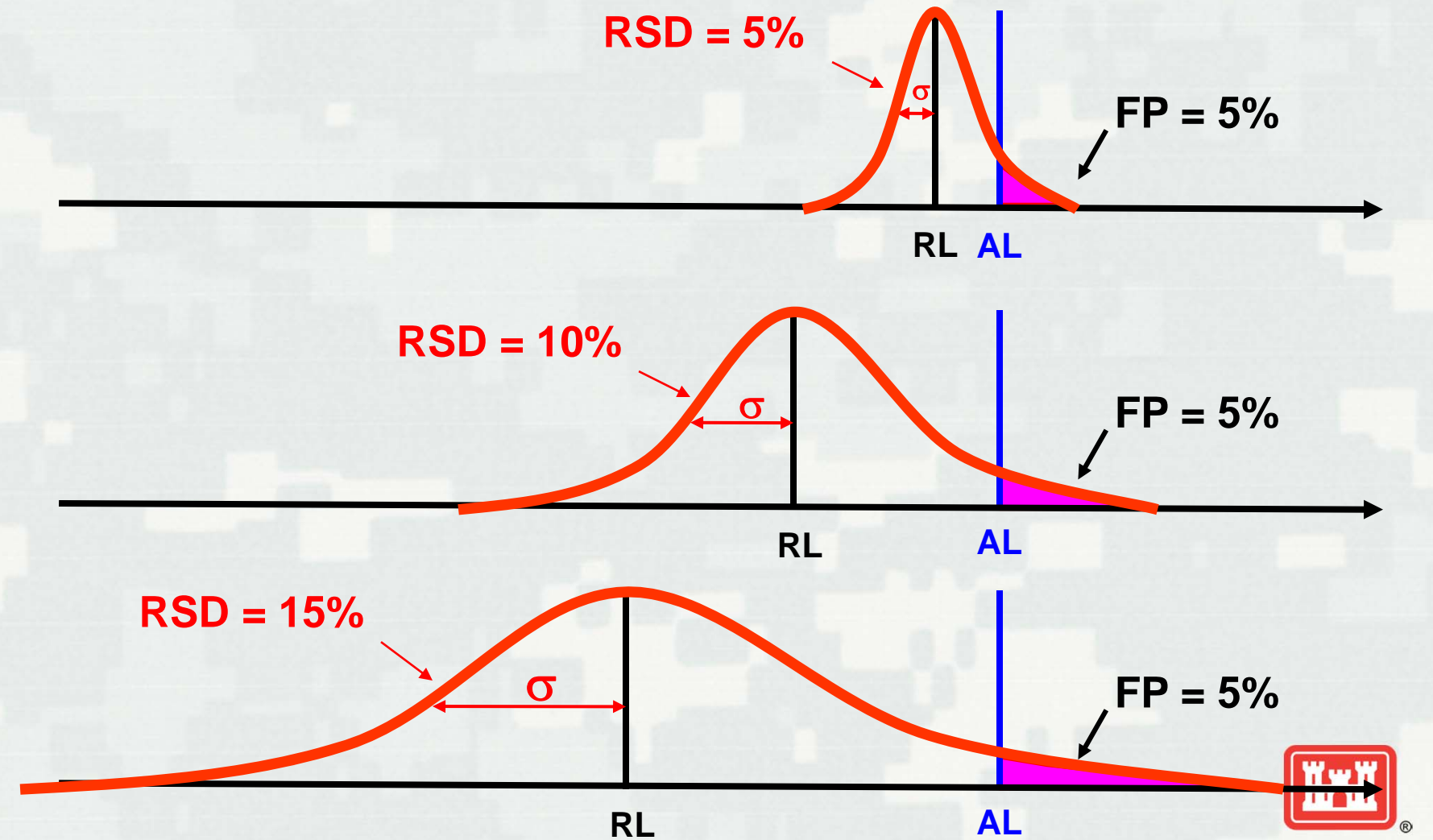


Need Quantitation at $RL < AL$

- To determine if $[X] < AL$, one needs to quantify $[X]$ at Project-Specific RL
- Project-Specific RL depends on:
 - **AL** (regulatory level, background, risk-based, etc.)
 - **Data Quality** (precision & bias)
 - **Tolerable Decision Errors** (FP/FN, Type I/II)
- $RL < AL$ but how much lower depends on **Data Quality** and **Tolerable Decision Errors**



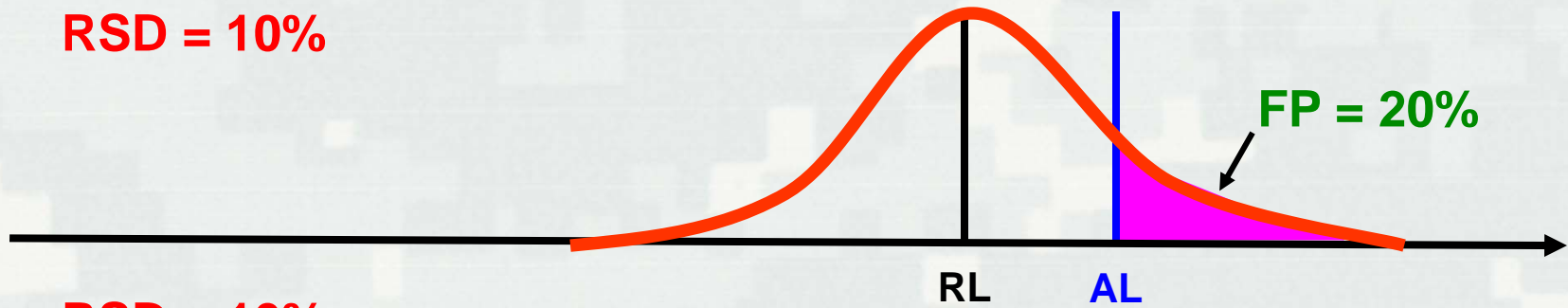
Data Quality – Precision



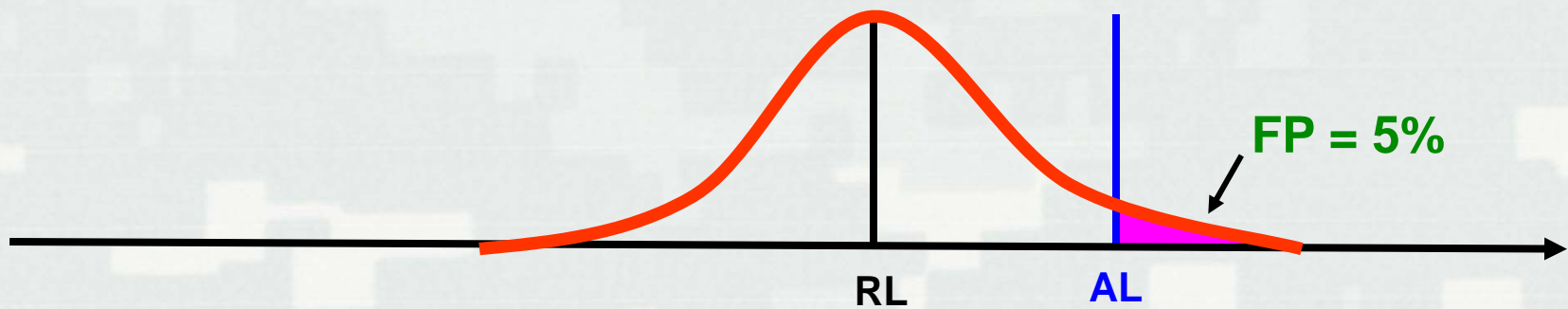
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Tolerable Decision Error – FP

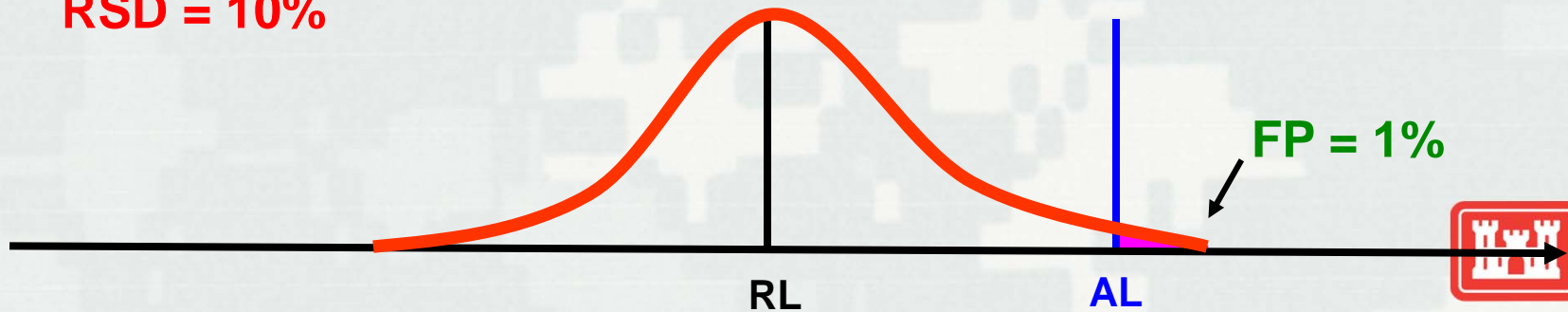
RSD = 10%



RSD = 10%



RSD = 10%



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RL vs. AL, DQ, & TDE

- Project-Specific RL depends on:
 - $RL < \underline{AL}$
 - $RL \downarrow$ if Data Quality (DQ) \downarrow
 - $RL \downarrow$ if Tolerable Decision Errors (TDE) \downarrow
- How to establish a Project-Specific RL?



How's RL Commonly Established?

- RL = Lab's LOQ (~ 80% projects) ?
- RL = Lab's MDL X
- RL = Regulatory Limit ?
- RL = AL X
- RL = $\frac{1}{2}$ AL ?
- RL = 3 – 5 times less than AL ?



Common Reasons for Lack of Project-Specific RL

- Don't know how
- Unknown data quality before analysis
- Huge field sampling errors
- Risk assessor establishes
 - PMs believe RL in number is adequate
- Limited resources
- Pandora's box



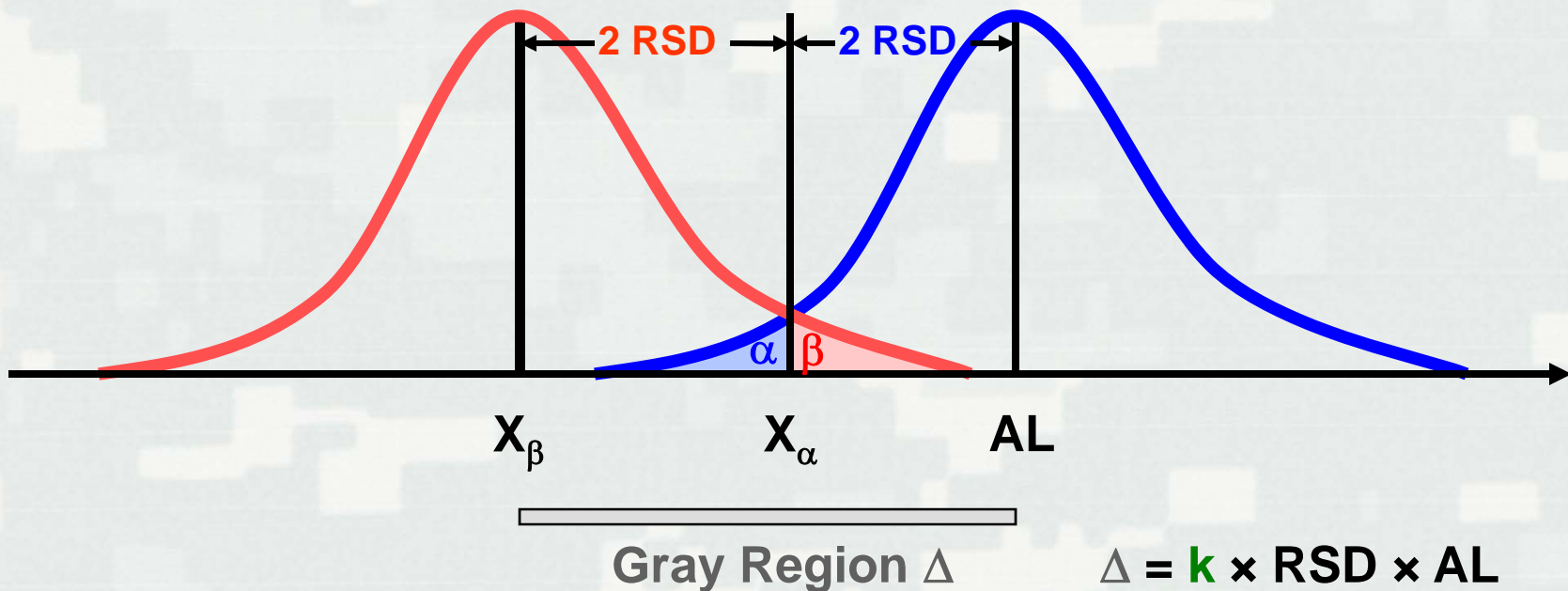
Inputs for Establishing Project-Specific RL

- **Action Level (AL)**: Regulatory level, background level, risk-based level, etc.
- **Anticipated Data Quality**: Based on DQOs, historical data, method, publication, etc.
- **Tolerable Decision Errors**: Type I/II (α/β) or FP/FN (Default $\alpha = \beta = 0.05$ or FP = FN = 5%)



Procedure for Establishing RL

$H_0: X > AL$, $H_A: X \leq AL$; decision errors $\alpha = \beta = 0.05$



$RL \leq (AL - \Delta) \times \%R = (AL - k \times RSD \times AL) \times \%R$, where

k = Error Factor, RSD = Rel Std Dev, $\%R$ = Recovery



Determine n, RSD, & %R

- $RL \leq (AL - k \times RSD \times AL) \times \%R$
 - $k = 4$ if $FP = FN = 5\%$ (2 RSD + 2 RSD)
 - $k = 5$ if $FP = 1\%$ (3 RSD) & $FN = 5\%$ (2 RSD)
 - $k = 6$ if $FP = FN = 1\%$ (3 RSD + 3 RSD)
- $RSD \leq [1 - RL / (AL \times \%R)] / k$
 - If $k = 4$, RSD must be $< 25\%$
 - If $k = 5$, RSD must be $< 20\%$
 - If $k = 6$, RSD must be $< 17\%$

k is based on tolerable decision errors

RSD & $\%R$ are data quality indicators at RL



Determine AL, DQ, & TDEs

- **Action Level (AL)**: Regulatory level, background, risk-based level, etc.
- **Anticipated Data Quality**: Based on DQOs, historical data, method, publication, etc.
 - Historical data
 - DoD QSM LCS Control Limits
 - Technology capabilities
- **Tolerable Decision Errors**: Type I/II (α/β)
 - Regulations
 - Practicality
 - Resources



Applications of RL

- **Select analytical methods**
 - RL vs. method performance
- **Select analytical labs**
 - RL vs. lab performance
- **Assess data quality**
 - Lab LOQ vs. project RL (LOQ \leq RL if compatible precision and bias)
- **Assess data usability**
 - Lab LOQ vs. project RL and AL (LOQ \leq RL $<$ AL if compatible precision and bias)



Case Study of Project-Specific RL


- Investigate ground water contamination by benzo(a)pyrene
 - Establish RL and associated precision & bias
 - Select analytical method & contract lab
- Inputs:
 - **AL**: 0.2 ppb (MCL)
 - **Anticipated Data Quality**: **RSD** = 9.5% & **%R** = 81% (DoD QSM LCS)
 - **Tolerable Decision Errors**: **FP** = **FN** = 5%
- $RL \leq (AL - 4 \times RSD \times AL) \times \%R = 0.10 \text{ ppb}$
 - if $RSD \leq 9.5\% \text{ \& } \%R \geq 81\%$



Case Study of Method & Lab Selections

- $LOQ \leq RL \leq (AL - 4 \times RSD \times AL) \times \%R = 0.10 \text{ ppb}$
if $AL = 0.2 \text{ ppb}$, $RSD \leq 9.5\%$ & $\%R \geq 81\%$

- DoD ELAP accredited Lab X:

 ➤ SW 8270: $LOQ = 0.2 \text{ ppb}$, $RSD = 16.2\%$ & $\%R = 82.5\%$
(Ideally, $LOQ \leq (0.2 - 4 \times 16.2\% \times 0.2) \times 82.5\% = 0.058 \text{ ppb}$)

- DoD ELAP accredited Lab Y:

 ➤ SW 8270: $LOQ = 0.2 \text{ ppb}$, $RSD = 9.8\%$ & $\%R = 84.5\%$

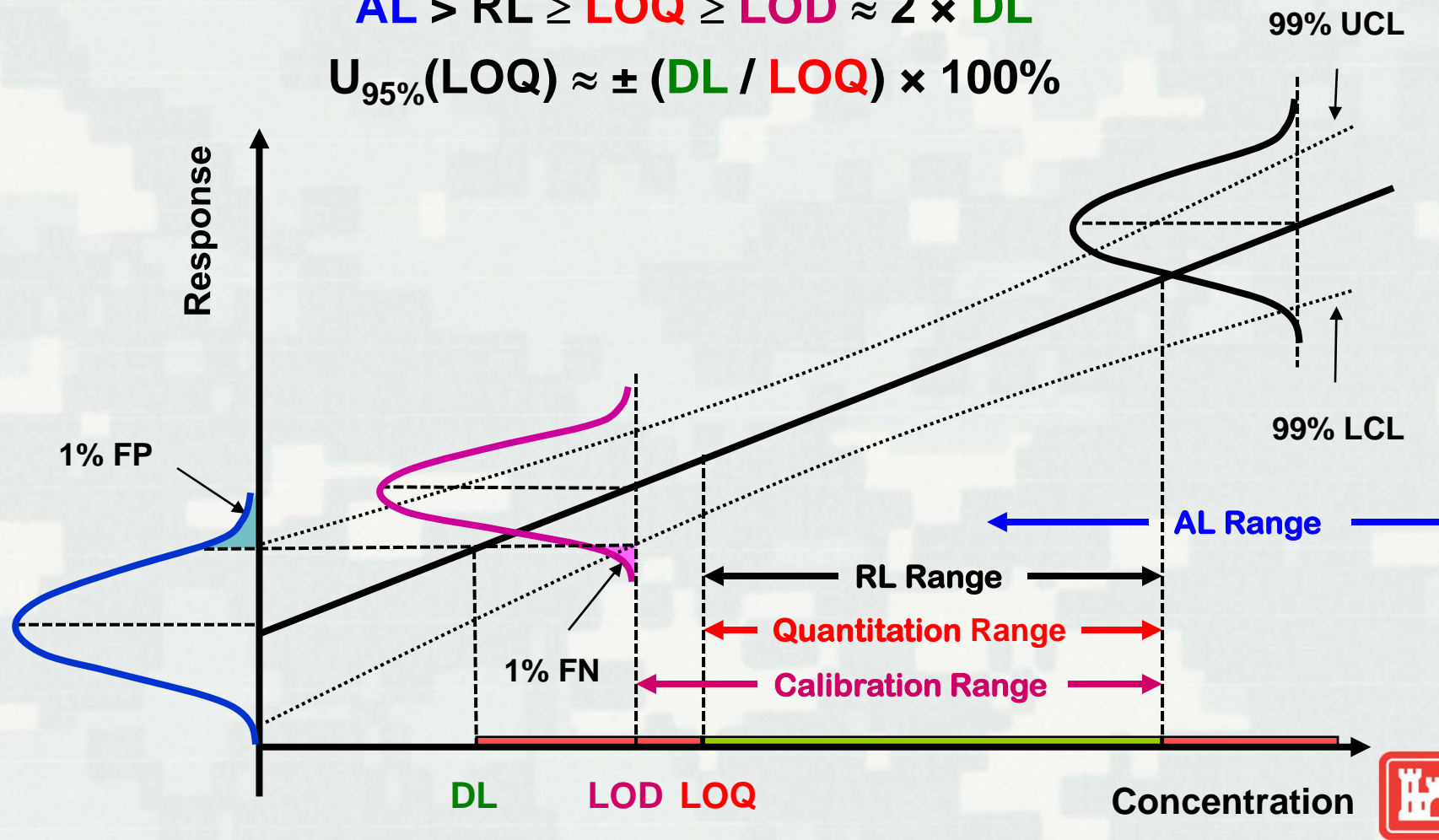
 ➤ SW 8310: $LOQ = 0.1 \text{ ppb}$, $RSD = 9.5\%$ & $\%R = 92.5\%$



Relationships among Various Limits

$$AL > RL \geq LOQ \geq LOD \approx 2 \times DL$$

$$U_{95\%}(LOQ) \approx \pm (DL / LOQ) \times 100\%$$



Major Issues

1. Old Rule of Thumb, $RL = 1/2 \sim 1/5 AL$, may not be adequate
2. $LOQ \leq RL$ is neither necessary nor sufficient; lower LOQ is not necessarily better – must consider precision & bias at LOQ and RL
3. RL based on precision & bias of clean matrix spikes is a minimum RL
4. Safety factors built in AL or risk assessment do not take care of decision errors
5. Huge field sampling errors trump lab analysis errors



Major Issue # 1

Old Rule of Thumb, $RL = 1/2 \sim 1/5 AL$, may not be adequate

If $AL = 100$, is $RL = 50 \sim 20$ adequate?

- Ideal SW-846 default, LCS CL = 70 – 130% (RSD = 10% & %R = 100%) and $\alpha = \beta = 0.05$ ($k = 4$)

$$RL \leq (AL - k \times RSD \times AL) \times \%R$$
$$\leq (100 - 4 \times 10\% \times 100) \times 100\% = 60$$

$$1/2 \sim 1/5 AL = 50 \sim 20 < 60 \quad \checkmark$$

- If LCS CL = 20 – 80% (RSD = 10% & %R = 50%)

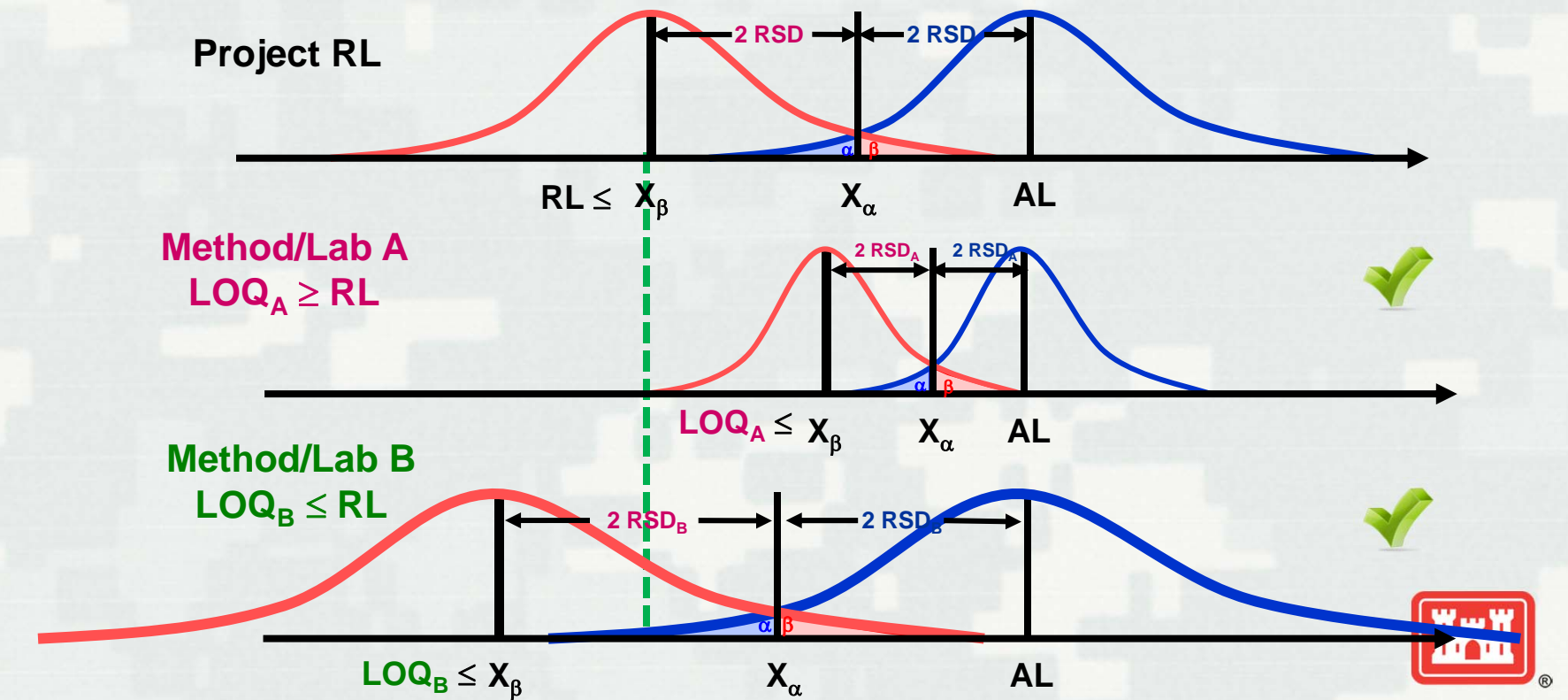
$$RL \leq (100 - 4 \times 10\% \times 100) \times 50\% = 30$$

$$1/2 AL = 50 > 30 \quad \times \quad 1/5 AL = 20 < 30 \quad \checkmark$$



Major Issue # 2

LOQ \leq RL is neither necessary nor sufficient; Lower LOQ is not necessary better – must consider P&B



Major Issue # 3

RL based on precision & bias of clean matrix spikes is a minimum RL

- Precision & bias based on clean matrix spikes such as method performance data, DoD QSM, or lab's LCS do not include matrix interferences
- A common denominator approach similar to DL, QL, and LCS studies for data comparability
- Because individual matrices are different, matrix-specific precision & bias cannot be reliably and cost-effectively estimated prior to sample analysis
- Matrix interferences may be assessed upon sample receipt or based on MS/MSD



Major Issue # 4

Safety factors built in AL or risk assessment do not take care of decision errors

- To have or not to have corrective action generally depends on the data quality and tolerable decision errors at the AL, not the accuracy of the AL
- Safety factors may not affect the decision rule, e.g., if $[X] > AL$, clean site; if $[X] \leq AL$, dirty site
- To reliably determine if $[X] > AL$, one needs to quantify and make the decision at RL



Major Issue # 5

Huge field sampling errors trump lab analysis errors

- Lab shall meet project MQOs, not DQOs. Lab is not liable for sampling errors and can't compensate large sampling uncertainty (Typical lab RSD must be $< 25\%$)
- Field sampling uncertainty cannot be reliably estimated without site investigation and is typically $> 4x$ lab uncertainty
- Lab performance yardsticks, method performance data, do not include field sampling uncertainty
- Need better sampling techniques (e.g., MIS) to reduce and control field sampling errors prior to evaluating field sampling uncertainty



Summary

- A simple, reliable, and practical procedure for establishing RL based on project-specific AL, anticipated data quality, and tolerable decision errors
- RL established based on precision and bias of clean matrix spikes is a minimum RL. More stringent RL is needed to cover sample matrix interferences and field sampling errors
- A useful tool for project planning, selecting analytical methods and contract labs prior to contract award
- $LOQ \leq RL < AL$ may not meet project DQOs



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Questions?

