DQOs and the Development of MQOs

Module 3

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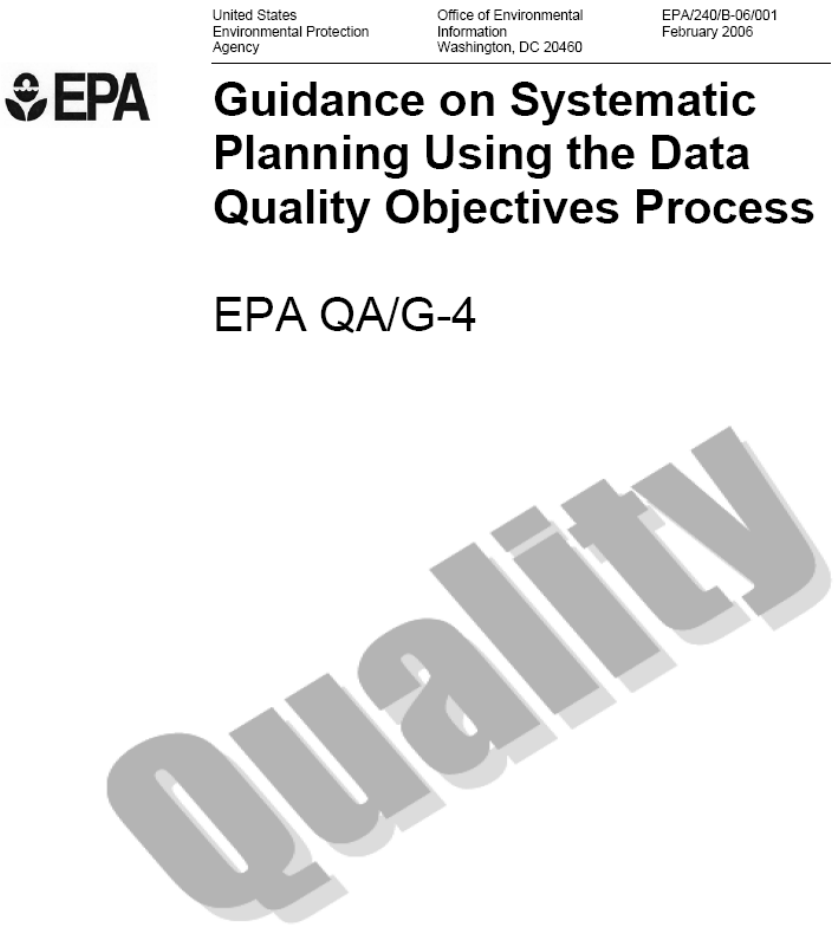
Data Quality Objectives (DQOs)

DQOs define performance criteria that limit probabilities of decision errors by:

- Considering the purpose of collecting the data
- Defining the appropriate type of data needed
- Specifying tolerable probabilities of decision errors

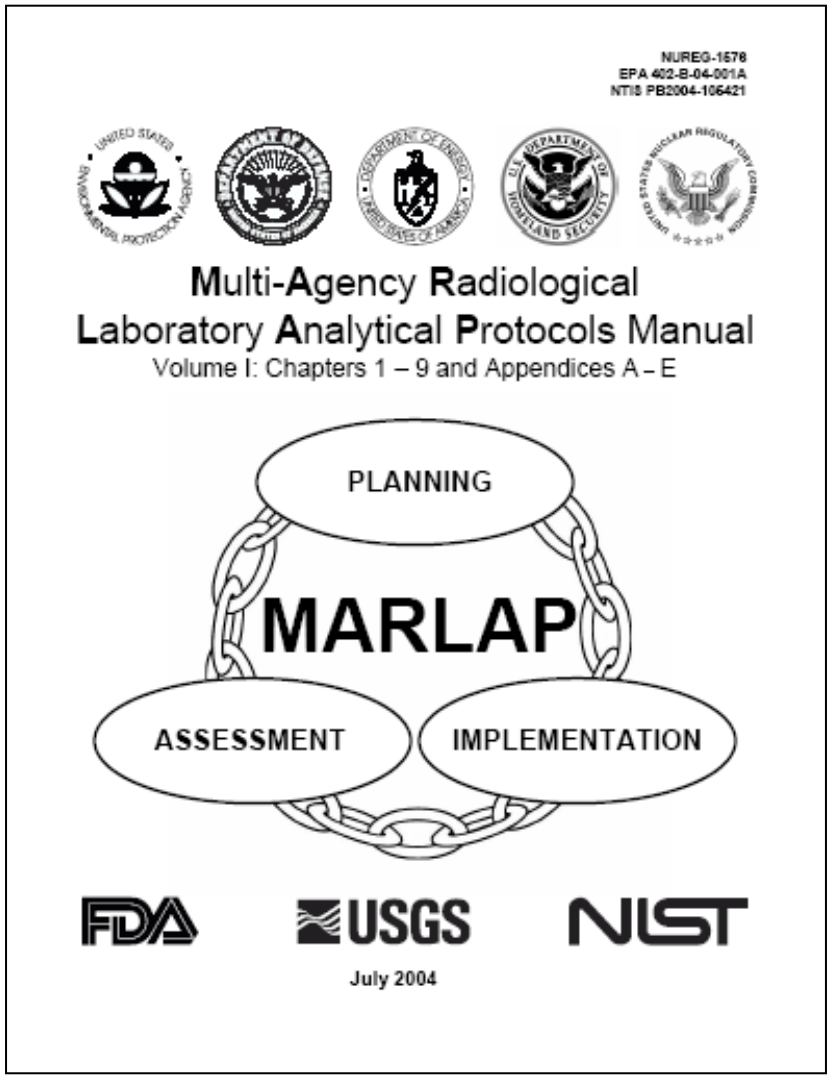


Measurement Quality Objectives (MQOs)

DQOs apply to both sampling and analysis activities

MQOs can be viewed as the analytical portion of the overall project DQOs

MQOs are the part of the project DQOs that apply to laboratory measurements and methods



MQOs (Continued)

MQOs are statements of objectives or requirements for analytical method performance characteristics. For example:

- Method uncertainty
- Detection capability
- Ruggedness
- Specificity
- Range

In a performance-based approach:

- MQOs are used initially for the selection and evaluation of analytical protocols
- MQOs are subsequently used for the ongoing and final evaluation of the analytical data

The primary MQO is the analytical measurement uncertainty at a specified concentration

Measurement Uncertainty

Measurement uncertainty:

"parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand." [GUM]

The uncertainty of a measurement can be expressed as an estimated standard deviation, called a ***standard uncertainty***

Evaluation of measurement data—Guide to the expression of uncertainty in measurement

JCGM 100:2008
GUM 1995 with minor corrections

https://www.bipm.org/en/publications/guides/

NIST Technical Note 1297
1994 Edition

Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results

Barry N. Taylor and Chris E. Kuyatt

Physics Laboratory
National Institute of Standards and Technology
Gaithersburg, MD 20899-0001

(Supersedes NIST Technical Note 1297, January 1993)

September 1994

The Action Level

- The ***action level*** (AL) is the analyte level (activity concentration) above which some action is believed to be necessary
- Given perfect information, you would take this action if and only if the true mean concentration exceeded the action level
- Unfortunately, your information is never perfect—there's always uncertainty due to:
  - Analytical measurement uncertainty
  - Sampling variability: variations among samples in time and/or space

Hypothesis Testing

- A hypothesis test chooses between a ***null hypothesis***, H_0, and an ***alternative hypothesis***, H_1
- H_0 is presumed true unless the data provide strong evidence for H_1
- Often H_0 is that the true mean concentration, μ, is $\geq$ AL
  - In which case, H_1 is $\mu <$ AL
- But H_0 can also be $\mu \leq$ AL
  - In which case, H_1 is $\mu >$ AL
- Sometimes H_0 is $\mu = 0$ and H_1 is $\mu > 0$
  - This is the familiar case of analyte *detection decisions*

Decision Errors

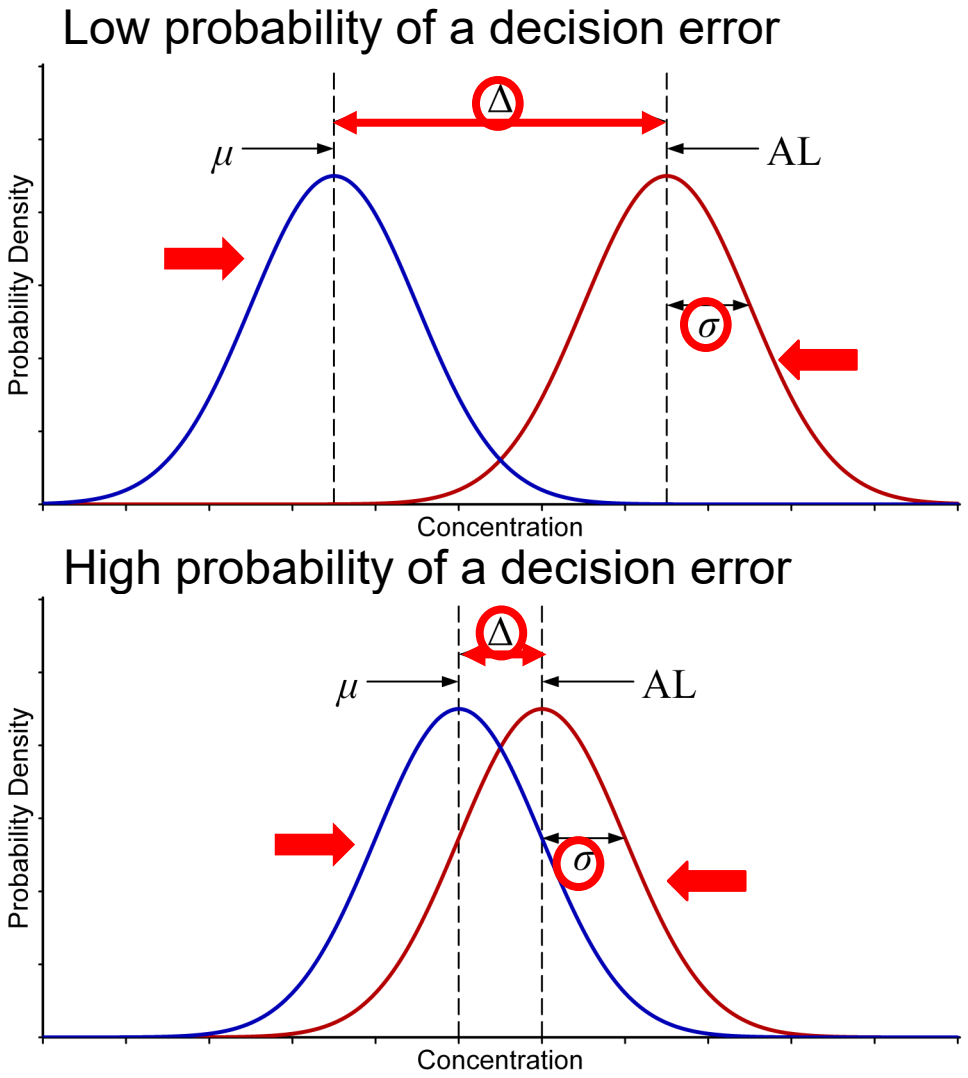
- Decision errors are possible because of uncertainty in the data
- ***Type I error*** = rejecting H_0 when it is true
  - Also called *false rejection*
- ***Type II error*** = failing to reject H_0 when it is false
  - Also called *false acceptance*
- Can't eliminate decision errors, just limit probabilities
- Choose a limit α for the probability of a Type I error
- Typically, α is small, in the range 0.01–0.10

Uncertainty and Decision Errors

- $H_0: \mu \geq AL$
- $H_1: \mu < AL$

The closer the mean is to the action level, the higher the probability of a decision error

Probability depends on Δ / σ



The Gray Region

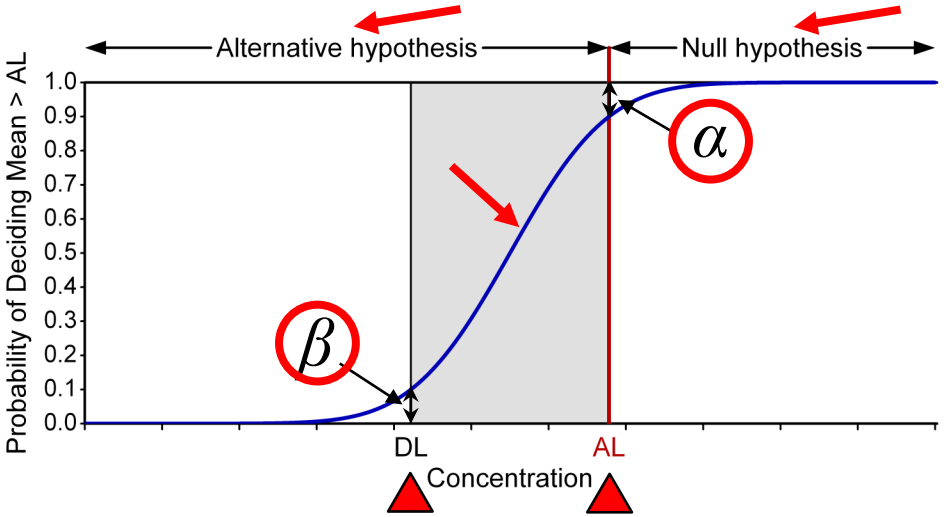
- *Choose* the maximum probability α for a Type I decision error (when H_0 is true)
  - Implement the hypothesis test to achieve the desired α
- Next problem: Control the probability of a Type II decision error (when H_1 is true)
- If H_1 is true but the mean concentration μ is near AL, the probability of a Type II error can be high because of uncertainty
- The range of concentrations where the Type II error probability is high is called the ***gray region***

The Bounds of the Gray Region

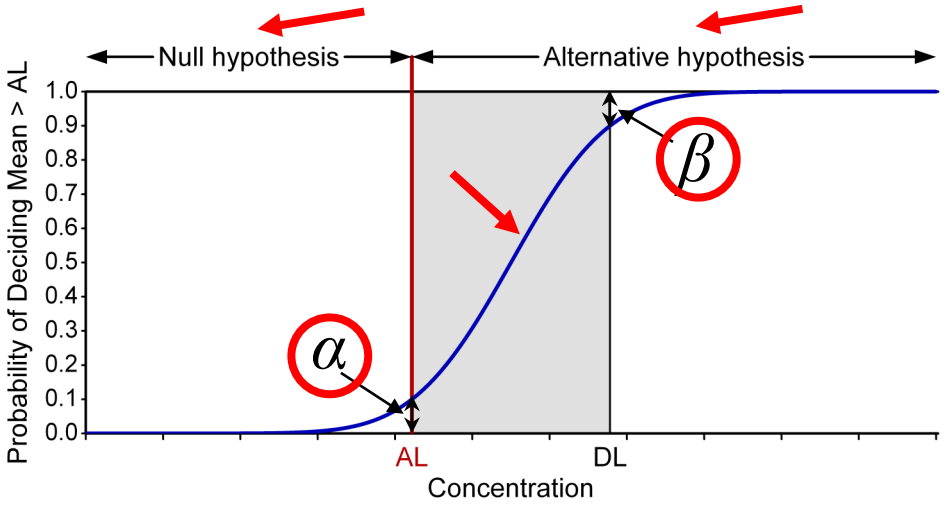
- The lower and upper bounds of the gray region are denoted by LBGR and UBGR
- One of these is the action level, AL
  - At the AL, the probability of a Type I error is limited to α
- MARLAP uses the term ***discrimination level*** (***DL***) for the "other bound of the gray region"
  - The DL is the concentration at which it is important to limit the probability of a Type II error, denoted by β
- **Note**: If AL = UBGR, then DL = LBGR, in which case DL should be the expected background concentration

The Gray Region Illustrated

- $H_0: \mu \geq AL$
  $H_1: \mu < AL$



- $H_0: \mu \leq AL$
  $H_1: \mu > AL$



Linking MQOs to DQOs

- To limit decision errors, the *total* uncertainty, σ, must be controlled
- From this goal, MARLAP derives limits for the analytical measurement uncertainty, σ_M
- These limits are the primary MQOs, with applications to:
  - Method selection and validation (MARLAP Chapter 6)
  - Evaluation of lab performance (Chapter 7)
  - Data validation (Chapters 7 & 8)

Method Uncertainty

- MARLAP defines ***method uncertainty*** to be the *predicted* measurement uncertainty obtained if the method were applied to a hypothetical laboratory sample with a specified analyte concentration (UBGR)
  - **Measurement uncertainty** is a characteristic of an individual measurement
  - **Method uncertainty** is a characteristic of the analytical method and the measurement process
- The ***required method uncertainty***, u_{MR}, is the maximum allowable method uncertainty

Two Scenarios

- Decisions about individual samples (as for bioassays)
- Decision about the mean of a sampled population (as for a MARSSIM final status survey)

- The scenario determines whether sampling variability is an issue
  - In Scenario 1, the analytical measurement uncertainty is the total uncertainty
  - In Scenario 2, both analytical measurement uncertainty and sampling variability are considered

Scenario 1: Decisions About Individual Samples

- In Scenario 1, the measurement uncertainty is the total uncertainty: $\sigma_M = \sigma$
- Require:

$$\sigma_M \leq \frac{\Delta}{z_{1-\alpha} + z_{1-\beta}}$$

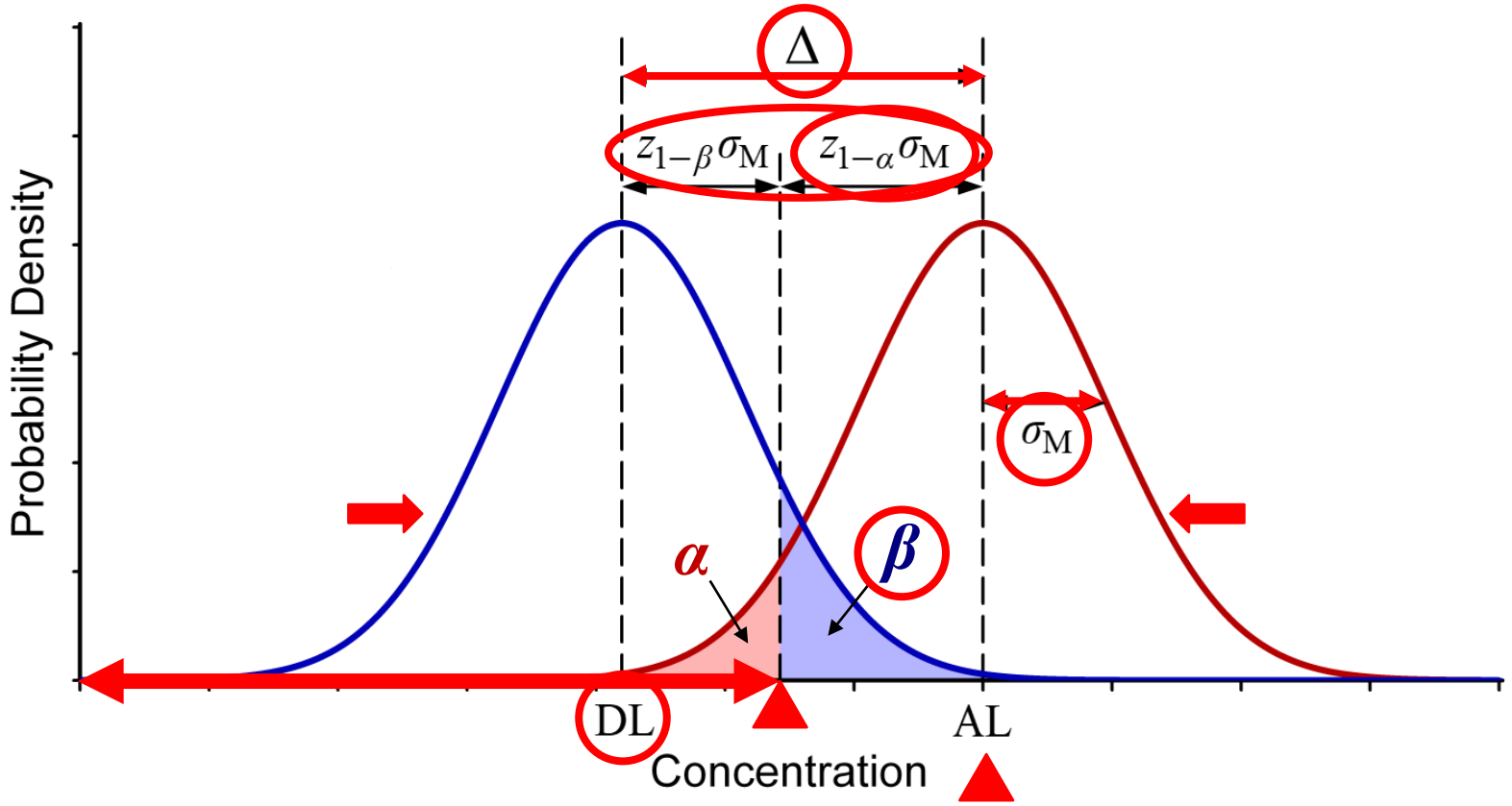
where:

  - σ_M is the measurement standard deviation at UBGR,
  - Δ is the width of the gray region, and
  - z_p, for any fraction p, denotes the $100p^{th}$ percentile of a normal distribution (e.g., $z_{0.95} = 1.645$)

First Case

- If AL = UBGR and DL = LBGR, require

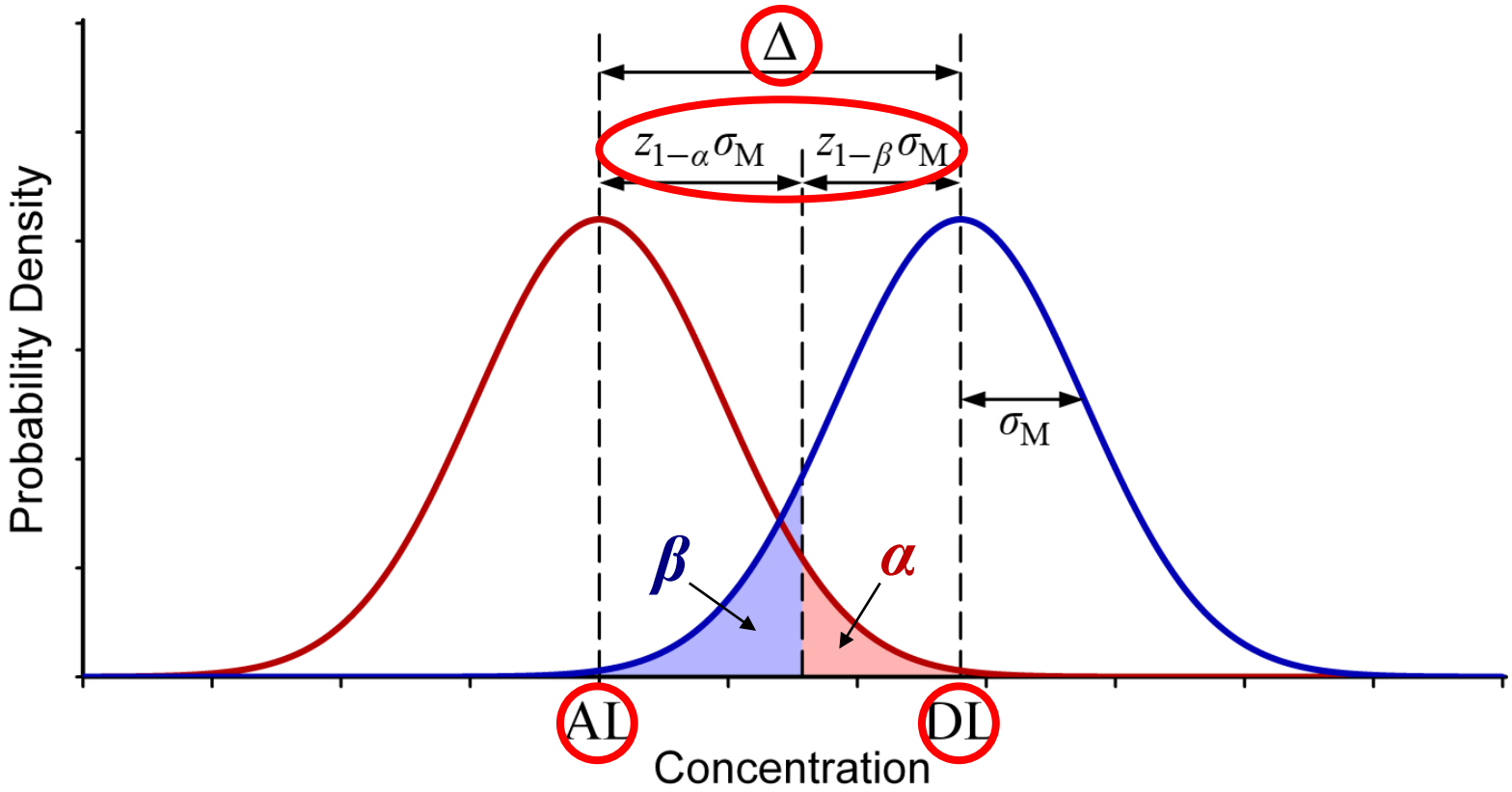
$$\mathrm{DL} + z_{1-\beta}\sigma_{\mathrm{M}} \leq \mathrm{AL} - z_{1-\alpha}\sigma_{\mathrm{M}}$$



Second Case

- If AL = LBGR and DL = UBGR, require

$$AL + z_{1-\alpha}\sigma_M \leq DL - z_{1-\beta}\sigma_M$$



Required Method Uncertainty for Scenario 1

- In either case, the requirement can be expressed as:

$$\sigma_M \le \frac{\Delta}{z_{1-\alpha} + z_{1-\beta}}$$

←***required method uncertainty, u_{MR}***

- For example, if $\alpha = \beta = 0.05$, then $z_{1-\alpha} = z_{1-\beta} = 1.645$, and

$$u_{MR} = \frac{\Delta}{z_{1-\alpha} + z_{1-\beta}} = \frac{\Delta}{1.645 + 1.645} = \frac{\Delta}{3.29}$$

- If $\alpha = 0.05$ and $\beta = 0.10$, then $z_{1-\alpha} = 1.645$, $z_{1-\beta} = 1.282$, and

$$u_{MR} = \frac{\Delta}{z_{1-\alpha} + z_{1-\beta}} = \frac{\Delta}{1.645 + 1.282} = \frac{\Delta}{2.93}$$

Required Relative Method Uncertainty

- MARLAP applies the required method uncertainty at any concentration below UBGR
- Above the UBGR, it applies the required *relative* method uncertainty, defined as:

$$\varphi_{MR} = \frac{u_{MR}}{UBGR}$$

- So, if the true concentration is < UBGR, the measurement standard deviation shouldn't exceed u_{MR}
- If the true concentration is > UBGR, the *relative* measurement standard deviation shouldn't exceed φ_{MR}

Detection Decisions

- Suppose LBGR = 0 and H_0 and H_1 are as follows:
  - H_0: Sample contains no activity ($\mu = 0$)
  - H_1: Sample contains activity ($\mu > 0$)
  - **Type I error**: Decide there is activity when there isn't
  - **Type II error**: Decide there is no activity when there is
- **This is the familiar framework for analyte detection decisions and MDC calculations**
- Since LBGR = 0, we have Δ = UBGR
- It's common to have $\alpha = \beta = 0.05$
- So, $z_{1-\alpha} = z_{1-\beta} = 1.645$ and $u_{MR} = \Delta / 3.29$

Required Detection Limit (MDC)

- Assuming the measurement standard deviation doesn't increase rapidly with concentration:

$$\text{MDC} \approx 3.29 \times \sigma_M$$

- We define $u_{MR} = \Delta / 3.29 = \text{UBGR} / 3.29$
- $\sigma_M \leq u_{MR}$ is nearly equivalent to $\text{MDC} \leq \text{UBGR}$

$$\sigma_M \leq \text{UBGR} / 3.29$$

$$\text{MDC} = 3.29 \times \sigma_M \leq \text{UBGR}$$

- Still specify u_{MR}, since MARLAP uses it to evaluate methods, laboratories, and data quality

Scenario 2: Decisions About the Mean of a Sampled Population

Decision Rule: If the true mean concentration in a survey unit is less than the action level, it may be released for unrestricted use. Otherwise, further remediation is required.

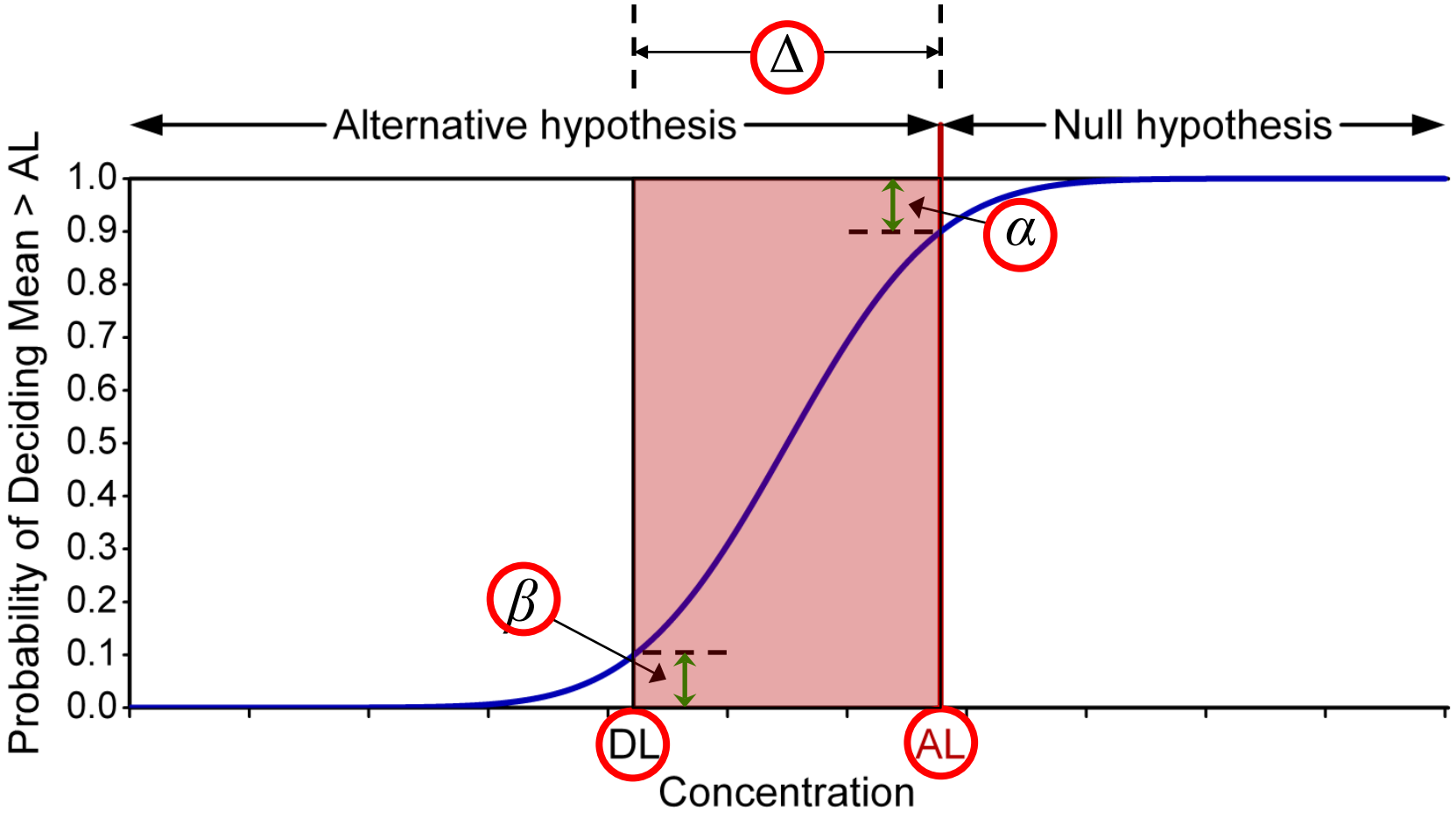
Choose null and alternative hypotheses (per MARSSIM):

H_0—The true mean μ exceeds AL

H_1—The true mean μ is below AL

AL = UBGR and DL = LBGR

Limiting the Total Uncertainty



To keep the number of samples reasonable, ideally, $\sigma \leq \Delta / 3$

Limiting the Method Uncertainty

Limit total uncertainty of the data so that $\sigma = \sqrt{\sigma_M^2 + \sigma_S^2} \leq \Delta / 3$

The sampling standard deviation, σ_S, depends on spatial and/or temporal variability of concentrations.

It's hard to control σ_S.

The analytical standard deviation, σ_M, is affected by laboratory sample preparation, subsampling, and analysis. It *can* be controlled.

Ideally, make σ_M small relative to σ_S: $\sigma_M \leq \sigma_S / 3$.

From $\sqrt{\sigma_M^2 + \sigma_S^2} \leq \Delta / 3$ and $\sigma_M \leq \sigma_S / 3$,

derive $\sigma_M \leq \dfrac{\Delta}{3\sqrt{10}} \approx \dfrac{\Delta}{10}$.

Then σ_M won't be a problem, although σ_S might be.

Required Method Uncertainty for Scenario 2

- The foregoing implies the required method uncertainty is:

$$u_{\mathrm{MR}} \approx \frac{\Delta}{10}$$

- Then the required *relative* method uncertainty is:

$$\varphi_{\mathrm{MR}} = \frac{u_{\mathrm{MR}}}{\mathrm{UBGR}}$$

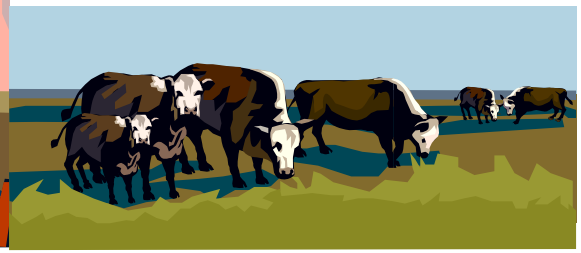
Required Quantitation Limit (MQC)

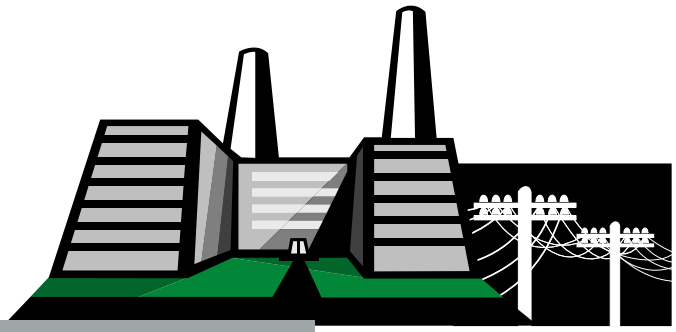
- Suppose LBGR = 0
- Then $u_{MR} = \Delta / 10 = UBGR / 10$ and $\varphi_{MR} = 0.10$
- Requiring $\varphi_{MR} \leq 0.10$ is the same as requiring that the *relative* standard deviation at UBGR be ≤ 10 %
- In other words, the *minimum quantifiable concentration* (MQC) should be no larger than UBGR
- So, the same requirement could be expressed in terms of either φ_{MR} or the MQC
- Still specify u_{MR} and φ_{MR}, since MARLAP uses them

Scenario 2: Example

Question: Does the milk from downwind cows have elevated concentrations of ^{90}Sr?

Downwind of Source





Potential Source of ^{90}Sr

Upwind of Source

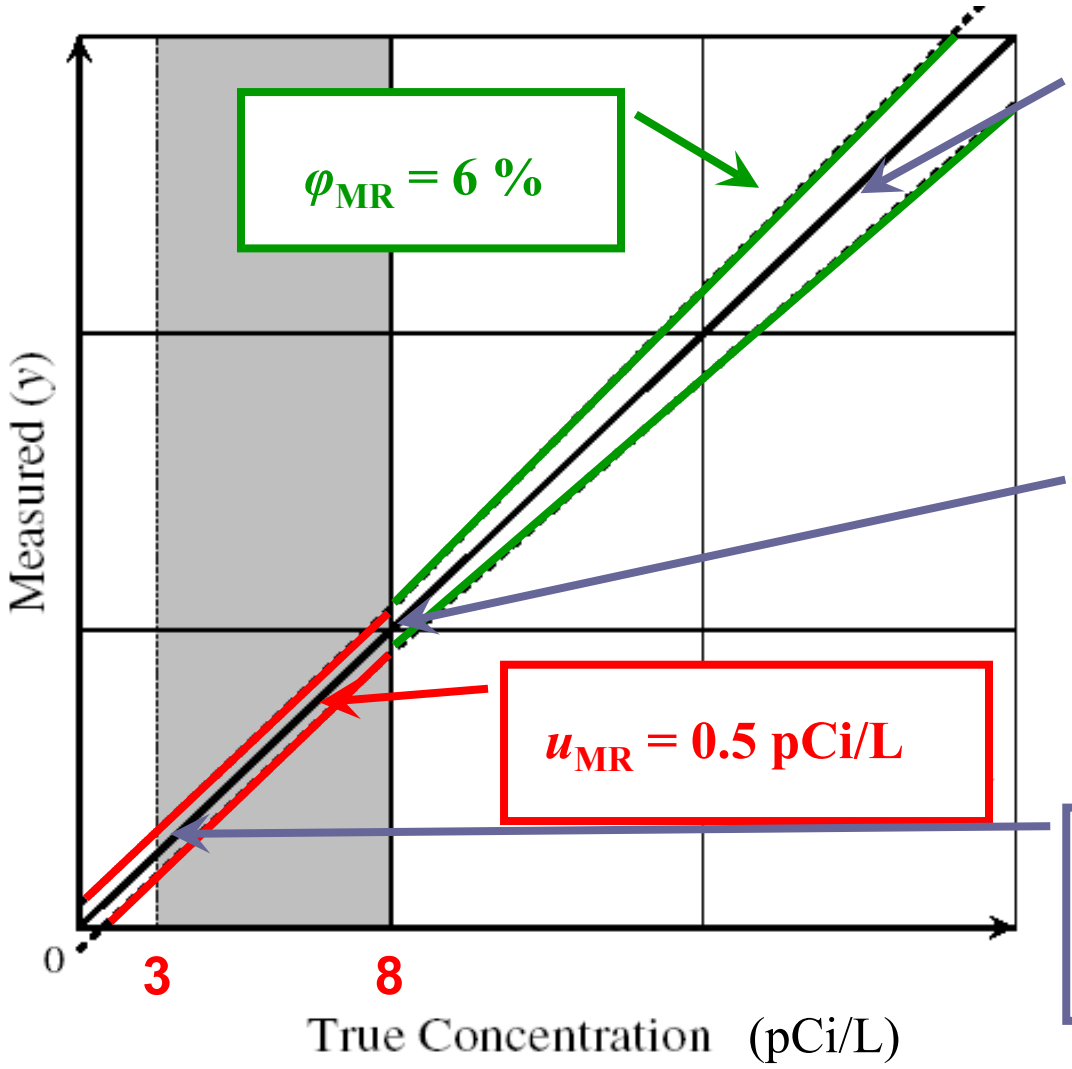
Calculate the Required Method Uncertainty

- Action Level: AL = 8 pCi/L ^{90}Sr in milk
- H_0: $\mu \geq$ AL and H_1: $\mu <$ AL
- Average background level: 2–3 pCi/L ^{90}Sr in milk
- Choose the Discrimination Level: DL = 3 pCi/L
- UBGR = AL = 8 pCi/L and LBGR = DL = 3 pCi/L
- Δ = (UBGR – LBGR) = 8 – 3 = 5 pCi/L

$$u_{MR} = \frac{\Delta}{10} = \frac{5\ \text{pCi/L}}{10} = 0.5\ \text{pCi/L}$$

$$\varphi_{MR} = \frac{u_{MR}}{\text{UBGR}} = \frac{0.5\ \text{pCi/L}}{8\ \text{pCi/L}} \approx 6\ \%$$

Required Method Uncertainty



Above UBGR (AL), the bound on the *relative* standard deviation is constant and equal to $\varphi_{MR} = u_{MR} / \mathbf{UBGR}$

The required method uncertainty, u_{MR}, is specified at UBGR

Below UBGR, the bound on the standard deviation is constant and equal to u_{MR}

SUMMARY: The Key to the MARLAP Process

In either scenario, the principal MQOs are defined by:

- The *required method uncertainty*, u_{MR}, below UBGR
- The *required relative method uncertainty*, φ_{MR}, above UBGR

$$\varphi_{MR} = u_{MR} / \text{UBGR}$$

For decisions about *individual samples*:

$$u_{MR} = \frac{\Delta}{z_{1-\alpha} + z_{1-\beta}}$$

For a decision about the *mean of a sampled population*:

$$u_{MR} = \frac{\Delta}{10}$$

Other Applications

- MARLAP applies the required method uncertainty, u_{MR}, and relative method uncertainty, φ_{MR}, to:
  - Method validation (see Module 8)
  - Evaluation of methods and laboratories (see Module 9)
  - Data verification and validation (see Module 10)