

# **Review and Reporting of Chemical of Concern (COC) Concentration Data Under the TRRP Rule (30 TAC 350)**

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# Data Review and Reporting: What Do You Need?/Where Do You Get It?



## TRRP Rule

- **Texas Register** (24TexReg 7413-7944): Sept. 17, 1999
- **TRRP Rule:** [www.tceq.state.tx.us/oprd/rules/indxpdf5.html](http://www.tceq.state.tx.us/oprd/rules/indxpdf5.html)

## TCEQ & EPA Guidance

- **TRRP Guidance** [www.tceq.state.tx.us/permitting/trrp.htm](http://www.tceq.state.tx.us/permitting/trrp.htm)
- **Review and Reporting of COC Concentration Data** (TRRP-13) December 2002
- **Assessment Planning** (TRRP-6) anticipated availability: Late 2004
- **Guidance for the Data Quality Objective Process for Hazardous Waste Sites** (EPA QA/G-4HW) [www.epa.gov/quality/qa\\_docs.html](http://www.epa.gov/quality/qa_docs.html)

**KEY POINT:** TRRP-13 effective February 1, 2003.

# Data Review and Reporting: *Why Do We Need RG-366/TRRP-13?*

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## *TRRP Rule*

30 TAC 350.54 Data Acquisition and Reporting Requirements

- (a) The person submitting data to the agency is responsible for the quality of the data.
- (b) The person shall provide data that are of sufficient and documented quality to meet the program and project objectives....

**KEY POINT:** TRRP-13 provides guidance for meeting rule requirements.

# Data Review and Reporting: 3 Steps

## 1 Laboratory

- **Data Review:** Compare lab results to method QC criteria.
- **Reporting:** Reportable data, lab review checklist, exception reports.

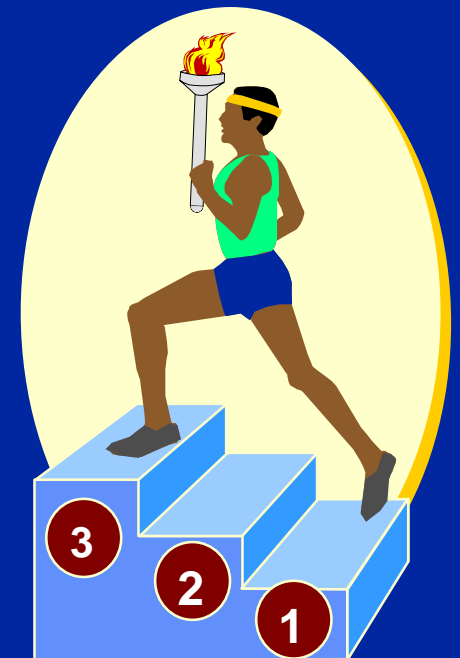
## 2 Person

- **Data Review:** Compare lab results to method and project QC criteria.
- **Reporting:** Data usability summary.

## 3 TCEQ

- **Review:** Verify that data reviews conducted, reports complete, and data usability justified.

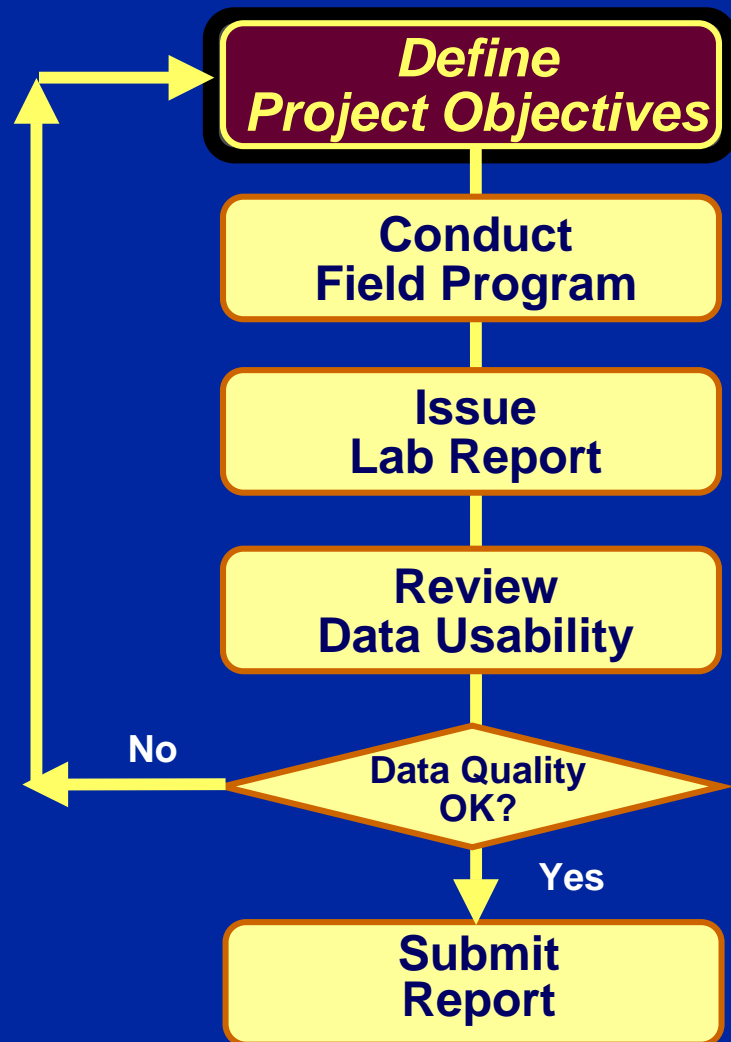
**Yippee !**



**WATCH  
OUT:**

Laboratory and method QC criteria may be different from **project-specific** DQOs.

# Data Quality: *Process Overview*



- Develop DQOs
- Get input from lab and/or TCEQ

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- Collect samples, field QC
- Analyze field parameters (e.g., pH, specific conductivity)

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- Analyze samples, review QC data
- Issue lab report

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- Conduct independent review of lab data (person or contractor).
- Determine if data meet project objectives.
- If needed, refine project objectives & collect additional data.

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- Incorporate data & submit report (e.g., APAR) to TCEQ

# Data Quality Objectives: Why Do We Need DQOs?

## TRRP Rule

30 TAC 350.54 Data Acquisition and Reporting Requirements

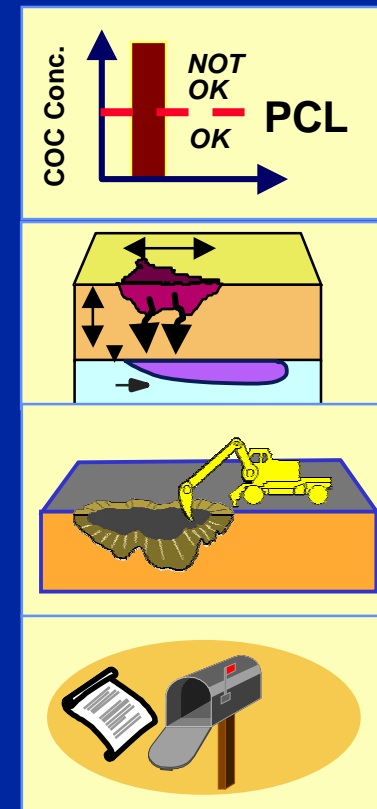
- (b) ...The project data quality objectives should be included in the APAR...
- (1) the rationale for the sampling design...
  - (2) the levels of required performance...
  - (3) the precision, accuracy, representativeness, comparability and data completeness objectives for the project.

## KEY POINT:

DQOs are needed in the report, but DQO plans do not need to be submitted for approval.

# “Critical Samples”

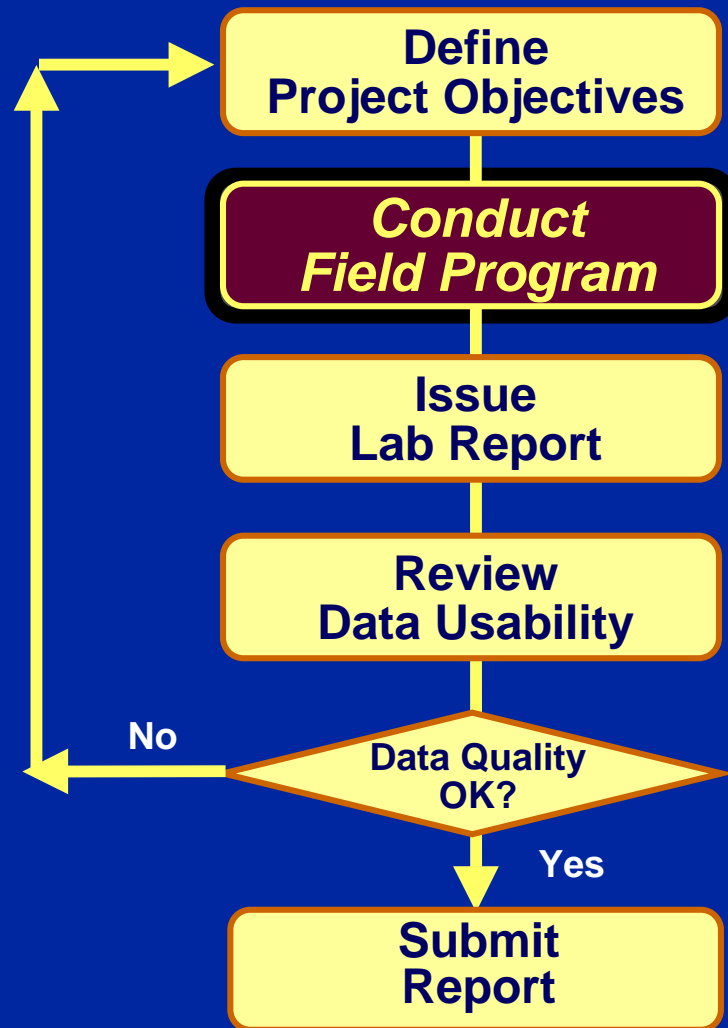
- **PCL:** Samples used to determine if a protective concentration level must be established for a COC per 30 TAC 350.71 (k)
- **Delineation:** Samples used to define lateral and/or vertical extent of affected media.
- **Attainment:** Samples used to demonstrate that response action is complete or that no further action is required.
- **Notification:** Samples used to determine if notification required per 30 TAC 350.55.



## KEY POINT:

Critical samples may be a subset of site samples.

# Data Quality: *Process Overview*



- Develop DQOs
- Get input from lab and/or TCEQ

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- Collect samples, field QC
- Analyze field parameters (e.g., pH, specific conductivity)

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- Analyze samples, review QC data
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# ***Field Analytical Program***

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- ***May be used to increase sampling density.***
  
- ***Examples of Common Field Analytical Methods:***
  - **Field Gas Chromatographs**
  - **Immunoassay Kits**
  - **X-ray fluorescence (XRF)**
  - **Gas Chromatography/Mass Spectrometry**
  - **Colorimetric Kits**
  
- ***Must be suitable for media and COC.***

**KEY POINT:** Supplements fixed laboratory data.

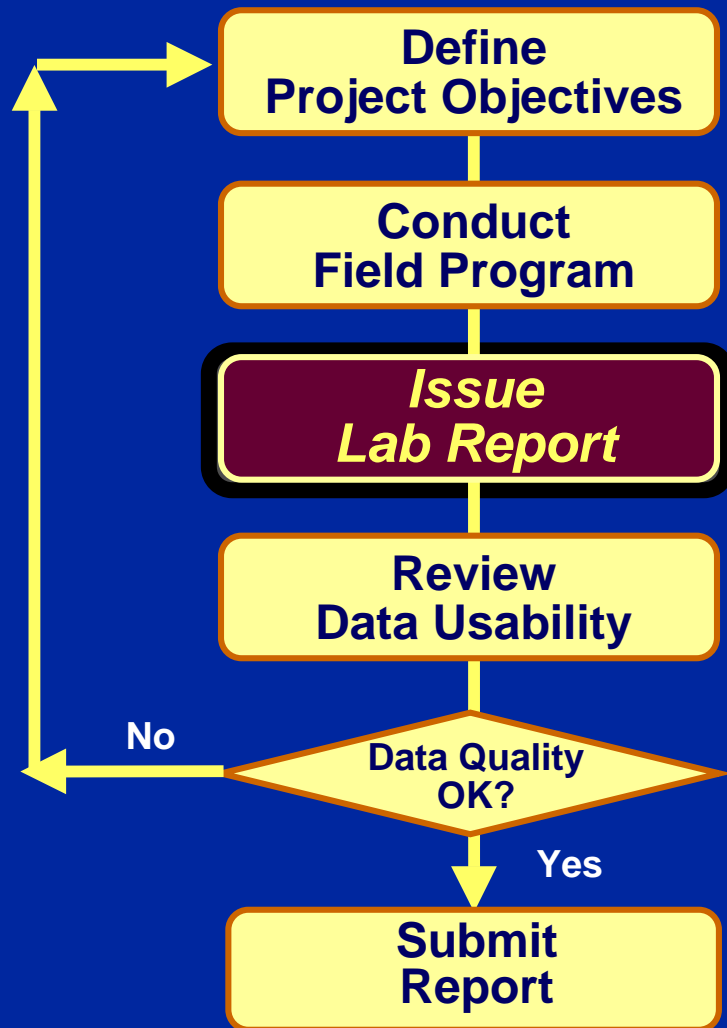
# ***Field Quality Control Program***

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- ***Negative Controls (e.g., blanks)***
- ***Positive Controls (e.g., spikes)***
- ***Confirmation Sample Analyses by Fixed Laboratory***
  - **Minimum 10%**
  - **Analyze samples with detections above the action level as well as samples with detections less than the action level**

***KEY POINT:*** Evaluate controls and compare field and fixed laboratory data to determine usability of field analytical data.

# Data Quality: *Process Overview*



- Develop DQOs
  - Get input from lab and/or TCEQ
- 
- Collect samples, field QC
  - Analyze field parameters (e.g., pH, specific conductivity)
- 
- Analyze samples, review QC data
  - Issue lab report
- 
- Conduct independent review of lab data (person or contractor).
  - Determine if data meet project objectives.
  - If needed, refine project objectives & collect additional data.
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- Incorporate data & submit report (e.g., APAR) to TCEQ

# Lab Heads Up: *Sample Administration*

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## *Moisture*

- Assign to all samples even if not specifically requested by client
  - Convert results to dry weight basis
- 

## *System Set-Up*

- MQLs & MDLs: Ensure current values in system prior to samples data input
  - Develop list of flags as necessary
- 

## *LORP*

- Obtain list of COCs from client
- Obtain PCL values from client

## **KEY POINT:**

Ensure that all laboratory personnel (analysts, project managers, etc.) aware that samples are for TRRP project.

# Lab Heads Up: *Reporting*

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- ***Laboratory Review Checklist:*** Complete on batch, project, or laboratory-defined basis and provide clear reference to associated samples. See TRRP-13 for example.
- ***Exception Reports:*** Include as needed. See TRRP-13 for example.
- ***Dry Weight:*** Provide results on dry-weight basis
- ***QC Limits:*** Include with sample reports or on separate report for surrogates, LCS data, MS/MSD data, and duplicate data, as applicable to the analytical method.

# Detection Limit Terms

Detection Limit (DL)

Estimated Quantitation Limit (EQL)

Contract Required Quantitation Limit (CRQL)

Limit of Detection (LOD)

Instrument Detection Limit (IDL)

Reliable Detection Limit (RDL)

Limit of Quantitation (LOQ)

Reliable Quantitation Limit (RQL)

Practical Quantitation Limit (PQL)

Reporting Limit (RL)

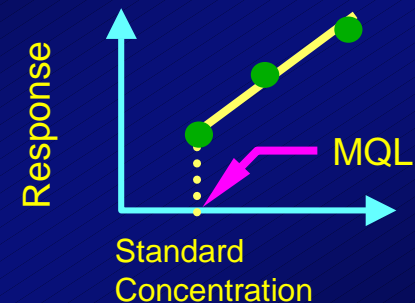
# TRRP Lab Testing: Key Definitions

TRRP 13  
2.1.3 (R9)

## Method Quantitation Limit (MQL)

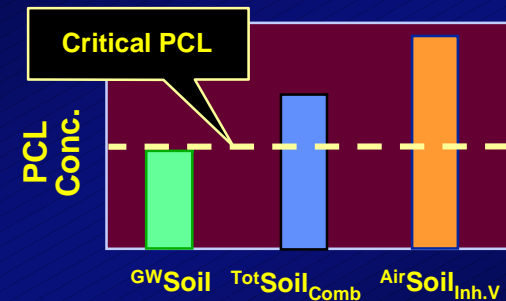
Lowest nonzero concentration on the calibration curve.

Lab Calibration Curve



## Critical PCL

- Lowest PCL for applicable exposure pathways.
- Level of required performance (LORP) for laboratory.



## KEY POINT:

When testing for TRRP COCs, be sure to use lab method with  $MQL < TRRP$  assessment level.

# Sample Quantitation Limit (SQL)

The method detection limit, as defined in [the rule], adjusted to reflect sample-specific actions, such as dilution or use of smaller aliquot sizes than prescribed in the analytical method, and takes into account sample characteristics, sample preparation, and analytical adjustments.

1. **Nondetected results are reported as less than the SQL - 350.54(h)(2).**
2. **May be used in lieu of MQL to demonstrate vertical delineation if COC cannot be measured to MQL and all reasonably available analytical technology has been used - 350.51(d)(1).**
3. **Includes requirement for reporting data on a dry weight basis after February 1, 2003.**

**KEY POINT:** The term, as used in the rule, is analogous to the sample-specific detection limit.

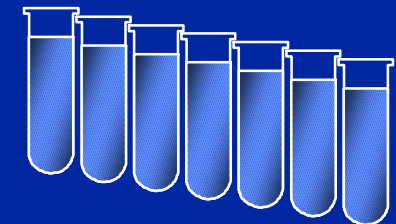


# TRRP Lab Testing: *Key Definitions*

## *Method Detection Limit (MDL)*

- Concentration at which a chemical can be measured with statistical confidence that concentration greater than zero
- Determined from replicate standards

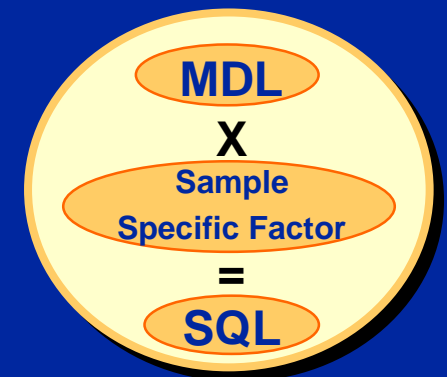
## *MDL Study*



Laboratory Replicates

## *Sample Quantitation Limit (SQL)*

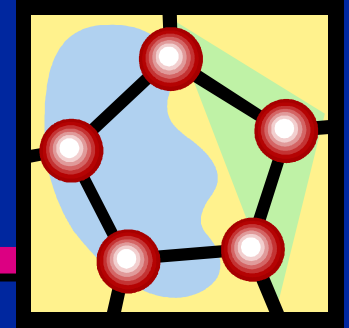
- For each COC in each sample, the SQL = the MDL adjusted for dilution, sample size, moisture content.
- Equivalent to “Sample Detection Limit”



## **KEY POINT:**

The lab must routinely check the MDL for each COC and verify ability to detect each COC at the MDL.

# Reasonableness Test



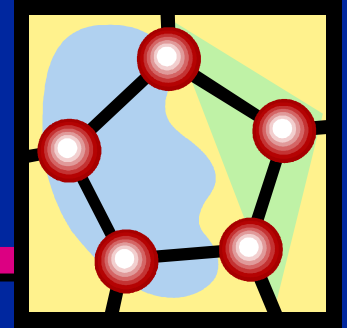
**30 TAC 350.54 (e)(4) ...The method detection limit should be routinely checked for reasonableness...**

- 1. Verifies the laboratory's ability to detect the COC at the MDL used for calculating SQLs.**
- 2. TRRP-13 provides a means for meeting the reasonableness requirement of the rule.**

***KEY POINT:* Detectability Check Sample (DCS).**

# DCS Requirements

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- 1. Reagent Matrix (aqueous and solid) spiked with the COC near to or within two to three times the calculated MDL and carried through the sample preparation and analytical procedures.**
- 2. Analyze quarterly during the period TRRP samples are analyzed.**
- 3. If the routine DCS supports the MDL, no additional MDL studies are necessary for TRRP.**

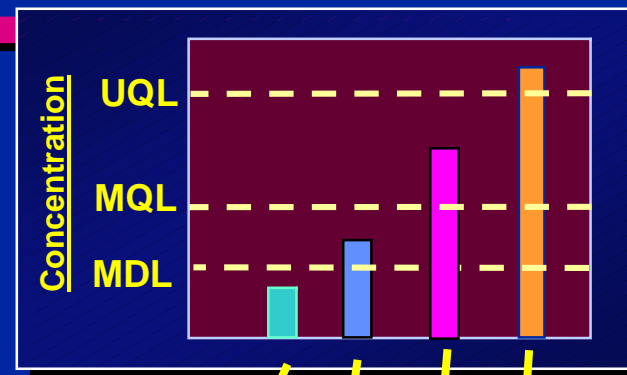
# Using the DCS

1. Analytical response must meet qualitative identification criteria in method or QAP.
2. If qualitative identification criteria not specified, laboratory must have confidence the response is different from the blank.
  - **If the COC is not detected in the DCS, the MDL is not supported.**
  - **The DCS concentration is adjusted until the DCS is detected.**
  - **After detection of the COC in the DCS, the laboratory uses the DCS concentration in lieu of the MDL to calculate SQLs.**
  - **Use the DCS concentration until a new MDL study establishes a reasonable MDL.**

**KEY POINT:** DCS documentation must be maintained by the laboratory.

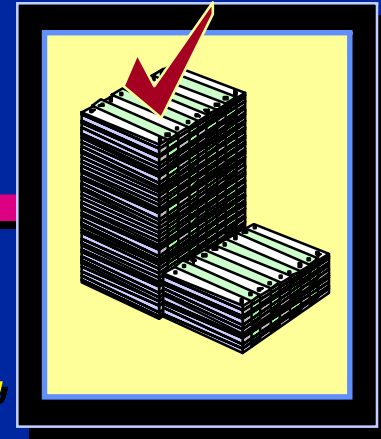
# Reporting the Results

<i>Instrument Response</i>	<i>COC Concentration</i>	<i>What Lab Reports</i>
$<MDL$	■ Non-detect	• “ND” or “<” or “U”
$>MDL$ & $<MQL$	■ Detected, but estimated	• Estimated conc. with “J” flag
$>MQL$ & $<UQL$	■ Detected and quantified	• Quantified conc.
$>UQL$	■ Detected, but estimated	• Estimated conc. with “E” flag



MDL = Method detection limit; MQL = Method quantitation limit;  
 SQL = Sample quantitation limit; UQL = Upper quantitation limit.

# Lab Report: *Required Contents*



- Reportable Data**
- **Results of sample analyses**
  - **Results of QC analyses (e.g., calibration, method blanks, instrument tuning)**
- 

- Laboratory Review Checklist**
- **Document Laboratory Data Review**
  - **Recommended format in TRRP-13**
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- Exception Report**
- **Document deviations from QAPP or SOPs**
  - **Describe QC failures and affected samples**

**KEY POINT:**

Rule requires data submittal, guidance recommends format.

# Lab Report: *Reportable Data*



- R1** Field chain-of-custody documentation
- R2** Sample Identification Cross-Reference
- R3** Test Reports
- R4** Surrogate Recovery
- R5** Laboratory Blank Data
- R6** Laboratory Control Samples (LCS)
- R7** Matrix Spike/Matrix Spike Duplicates (MS/MSD)
- R8** Laboratory Duplicate
- R9** Method Quantitation Limits (MQLs)
- R10** Other Problems or Anomalies

# Completing LRCs and ERs

- ***Signature Page:*** Laboratory Manager or designee signs release statement. Note: page will be modified to include laboratory report numbers. Please do this now.
- ***LRCs for Reportable Data:*** Review for consistency with reportable data prior to release of data package.
- ***LRCs for Supporting Data:*** Completed by personnel with knowledge of analytical conditions under which samples were analyzed.
- ***Exception Reports:*** Reference pages of analytical report. Example: Volatile MS/MSD outside laboratory acceptance criteria as shown on pages X through Y instead of listing each compound.

## **KEY POINT:**

Errors in LRCs for reportable data reduce data users confidence in laboratory's performance and will generate requests for additional information.



# Potential Extras: *TICs*

## *Tentatively Identified Compounds*

- ***When to Report***  
Submit if analysis of TICs by GC/MS requested by TCEQ; requirement details in TRRP-10
- ***What to Report***  
Where possible, for each TIC, provide CAS number, chemical name, retention time, estimated concentration; description will be amendment to TRRP-13



*Tetra-ethyl-watchamacallit*

# Lab Report: *Supporting Data*

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- S1** Initial calibration (ICAL)
- S2** Initial and continuing calibration verification (ICV and CCV)  
Continuing calibration blank (CCB)
- S3** Mass Spectral Tuning
- S4** Internal Standard Areas
- S5** Raw Data
- S6** Dual Column Confirmation
- S7** Tentatively Identified Compounds (TICs)
- S8** Interference Check Sample (ICS) Results
- S9** Serial dilutions, post digestion spikes, method of standard additions
- S12** Standards Documentation

# Lab Report: *Supporting Data for Audits*

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- S10** Method Detection Limit (MDL) Studies (and associated DCS results)
- S11** Proficiency Test Reports
- S12** (Moved to previous slide)
- S13** Compound/Analyte identification procedures
- S14** Demonstration of Analyst Capability (DOC)
- S15** Verification/validation documentation for methods
- S16** Laboratory Standard Operating Procedures

# Summary

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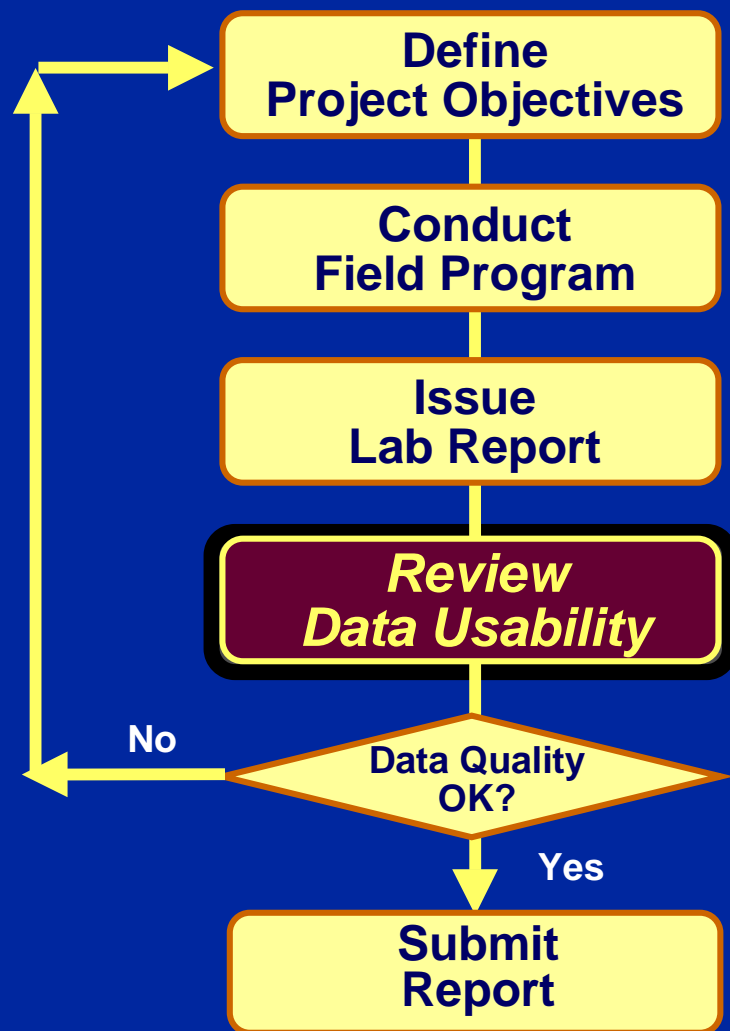
*Person  
Provides*

- **Project Objectives**
  - **COCs (suggest using CAS No.)**
  - **LORPS**
  - **Identification Critical Samples**
- 

*Laboratory  
Provides*

- **Appropriate methods**
- **Laboratory acceptance criteria**
- **Deliverables**

# Data Quality: *Process Overview*



- Develop DQOs
- Get input from lab and/or TCEQ

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- Collect samples, field QC
- Analyze field parameters (e.g., pH, specific conductivity)

---

- Analyze samples, review QC data
- Issue lab report

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- Conduct independent review of lab data (person or contractor).
- Determine if data meet project objectives.
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---

- Incorporate data & submit report (e.g., APAR) to TCEQ

# Data Usability

## 30 TAC 350.54

### *TRRP Rule*

- (b) The person shall provide data that are of sufficient and documented quality to meet the program and project objectives...
- (f) The person shall identify any data that may be affected by laboratory deviations from the analytical method or by the laboratory's performance not meeting project-required and/or method-required quality control acceptance criteria. The person shall also identify any data that may be affected by improper field procedures.

### **KEY POINT:**

**Data Usability Summary is suggested format for meeting requirements.**

## TRRP-13: Data Reporting and Review

# Data Usability Review: *What to Check*

- **Field to Lab:** Holding times, preservation, sample containers, sample collection procedures
- **Lab Control Samples:** LCS Recoveries, LCS duplicate recoveries, LCS/LCSD precision.
- **Matrix Spikes:** MS Recoveries, MSD recoveries, MS/MSD precision.
- **Surrogates:** Percent recoveries for organics.
- **Lab Duplicates:** Percent recoveries for inorganics.
- **MQLs and SQLs:** Compare to level of required performance.
- **Supporting Data:** Check the results of lab's review on LRC



### **KEY POINT:**

Guidance provides optional forms for summarizing results of data usability review.

# Laboratory Reporting: **Common Errors**

- **SQLs**
  - Not provided or SQL labeled as MDL
  - Incorrectly calculated
  - LIMS uses aqueous MDLs for soil data
- **MQLs**
  - Laboratory does not indicate if MQLs are adjusted.
  - Laboratory does not submit unadjusted MQLs.
- **COCs**
  - Analytical staff do not communicate to project manager which compounds are not spiked into LCS.
- **LRCs**
  - Laboratory incorrectly checks “Yes” even though quality control report indicates data outside acceptance criteria.
  - Laboratory does not specifically state which COCs not spiked into LCS; laboratory uses generic statements.
- **Others**
  - Data column headings do not match regulatory nomenclature; i.e. PQL instead of MQL

## **KEY POINT:**

Have experienced personnel review TRRP reports.



# Data Usability Review: *Comparison of LRC and DUS Acceptance Criteria*

- **Laboratory** Laboratory ONLY completes exception report if recoveries and/or precision do not meet laboratory acceptance criteria.
- **Data Reviewer** Data reviewer qualifies COC data based on project acceptance criteria developed during planning process. Intended use of data must drive acceptance criteria (see EPA QA/G-4HW and Introduction to Data Quality Indicators [<http://www.epa.gov/quality/trcourse.html>])
- **TRRP-13 Appendix D** Example Tool: project objectives determine appropriate acceptance criteria.

**KEY POINT:** Data reviewer applies criteria developed during planning process. TRRP-13 does NOT specify acceptance criteria.

# Data Usability Review: *Data Qualifiers & Codes*

## DATA QUALIFIER

**U**

**J**

**UJ**

**NJ**

**R**

## WHAT IT MEANS

- ***Not Detected:*** Analyte not detected above SQL.
- ***Estimated:*** Analyte detected, qualitative identification criteria met, but concentration is approximate.
- ***Not Detected:*** Analyte not detected, but the SQL is estimated.
- ***TIC:*** Tentatively identified compound.
- ***Rejected:*** Data are unusable due to QC problems.

## Bias Code

**H**

**L**

## WHAT QC DATA MEANS

- ***High:*** Lab result could be higher than actual value.
- ***Low:*** Lab result could be lower than actual value.

# Data Usability Summary

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## ***Submittal***

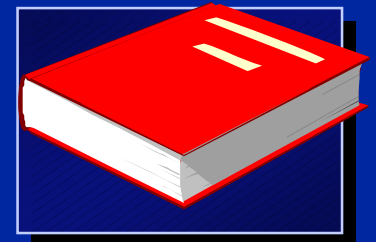
Include as appendix to TRRP-required reports having laboratory data (e.g., APAR, RACR, RAER)

## ***Contents***

Results of Data Usability Review and supplemental review, if conducted

## ***Format***

Use text, tables, figures, and/or checklists as needed to summarize findings of Data Usability Review and justify any data flags or qualifiers



### ***KEY POINT:***

Guidance does not specify format; so use presentation most convenient to convey results of DUS.

# Data Usability Summary (DUS)

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*Information  
to  
include  
in the  
DUS*

**Intended Use of Data**

**Samples Cross-Referenced to Laboratory  
identifications**

**Analytical Parameters and Method References**

**Field Data Reviewed and Summary of Findings**

**Quality Control Data Reviewed and Summary  
of Findings**

**Supplemental Data Review from LRC/ER**

**Review criteria (acceptance criteria) –**

**Laboratory, project, method, TRRP-13**

# Data Usability Summary (DUS)

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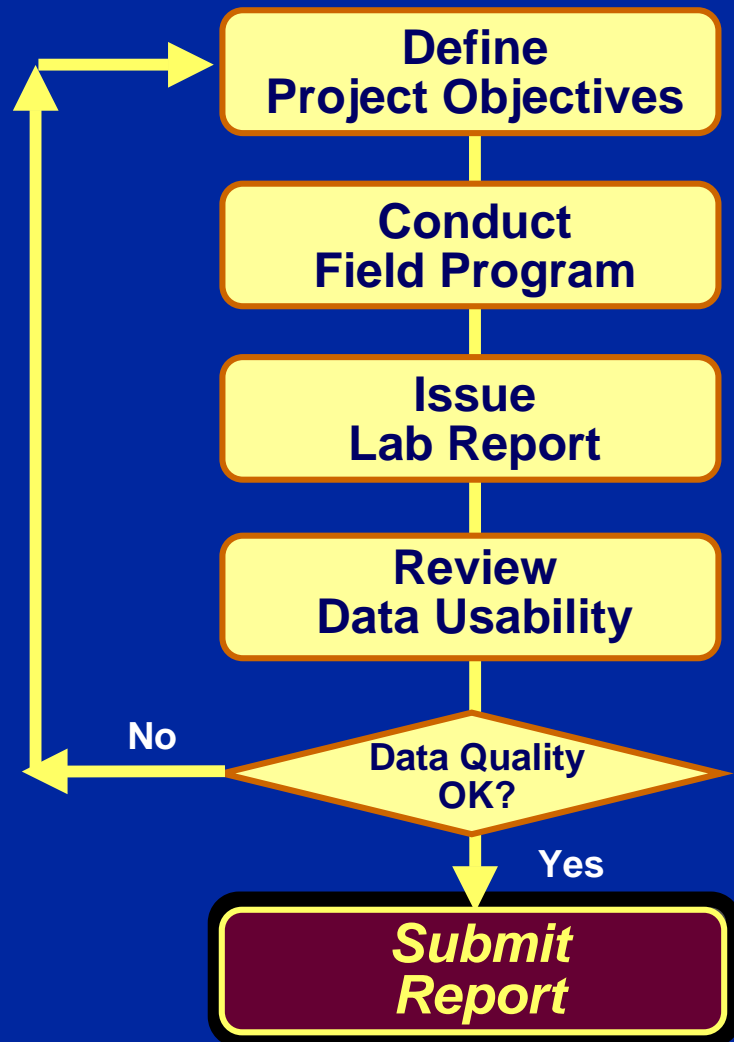
## *Important Points to Cover*

1. List specific samples and constituents that did not meet acceptance criteria.
2. List qualifiers/bias codes and rationale for qualifiers/bias codes for each sample/analyte.
3. Determine and describe usability of data.
4. Discuss technology (e.g., alternate methods, sample dilutions) used to reduce matrix interference or to achieve SQLs at or below PCLs

### **KEY POINT:**

Provide conclusions about usability of qualified data.

# Data Quality: *Process Overview*



- Develop DQOs
- Get input from lab and/or TCEQ

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- Collect samples, field QC
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- Analyze samples, review QC data
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- Incorporate data & submit report (e.g., APAR) to TCEQ

# Problems Commonly Observed by TCEQ

- Test reports do not include the SQL or the dilution factor.
- Unadjusted MQLs are missing.
- Explanation for dilutions when all method analytes are reported as not detected is not included in the report.
- It appears that laboratories are not reviewing data; just completing the LRC without review of the report. This is based on inconsistencies between the reported data and the LRC. This is bad.
- Data reviewers do not identify discrepancies between reported data and the LRCs. This is worse.

**Questions???**