Standard Operating Procedure for Reporting Qualified Data and Correcting Quality Control Problems

1.0 SCOPE

This SOP is intended to provide guidance to analysts and managers for qualifying data when quality control (QC) results are outside established control limits and re-analysis is not a viable or desired option. It provides guidance to lab analysts on determining the root cause(s) of QC failures using a systematic approach to diagnose and fix problems that caused the QC failure. This SOP also specifies other conditions under which data qualifier flags are associated with reported results.

2.0 REPORTING QUALIFIED DATA

Appropriate data qualifier flags as specified in Chapter 62-160, Florida Administrative Code (Appendix 1) must be used to qualify results. The following examples of conditions leading to data qualifications are provided, however these examples are not intended to be exhaustive; additional details concerning data qualified under specified scenarios may be found in method SOPs such as Taxonomy. Data qualifiers may be applied whenever necessary:

- Holding times are not met;
- Samples were improperly preserved;
- Samples were improperly collected;
- Continuing Calibration Verification is outside control limits;
- Surrogate spike recovery is outside control limits;
- Matrix spike recovery is outside control limits;
- Analytical precision for results above quantitation limits is outside control limits;
- Analyte concentrations are below method detection limits;
- Analyte concentrations are below practical quantitation limits but above method detection limits;
- Analyte concentrations (as analyzed) fall outside the calibration range;
- Interferences are evident that affect accuracy or precision of analytical data;
- Random contamination or reagent contamination is evident at a level that affects accuracy;
- Laboratory Control Sample (LCS) recovery is outside control limits;

Appendix 2 contains flow charts that outline the decision making process for qualifying data as estimates whenever valid QC failures are observed. Appendix 3 provides step-by-step instructions on addressing LCS failures in multi-component analyses. Comments and/or non-conformance reports are affixed to reported test method results whenever data qualifiers are

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indicative of conditions that may have the potential to cause adverse impacts to data quality. Additional guidance for specific conditions is provided below.

Initial Calibration Verification (ICV) or Continuing Calibration Verification (CCV)

Ideally, all ICV and CCV recoveries should fall within methodological limits for acceptability. In practice however, achieving those requirements for all analytes can be challenging for multi-component test methods. To address those challenges, accreditation requirements state that, unless required by method or regulation, methods employing an internal standard require calibration verification only at the beginning of each analytical batch, not at the end of the analytical batch. ICV and CCV results shall be first evaluated against individual method requirements. If the results do not meet method requirements but the associated analytical results are below the detection limit or below the threshold for quantitation and no systematic bias is observed, an appropriate test comment and/or result qualifier (J) shall be provided to indicate the ICV or CCV is outside control limits. However, when a systematic bias or system failure is detected or the affected analytical data are above the quantifiable threshold, the analysis shall be re-run if possible. If re-analysis is not an option, the analytical results shall be qualified as estimates.

Matrix Spikes

Data may be qualified should matrix spike recovery fall outside control limits. Spike levels used to assess recoveries should be targeted to approximate levels of analyte expected in the sample. Excessively high spike levels relative to sample analyte concentrations can make recovery appear more optimistic than it might actually be at the level present in the sample. Conversely, whenever sample analyte levels (background concentrations) exceed spike levels, accurate assessment of recovery can be difficult or impossible. Therefore, use the following guidelines to determine whether to qualify data.

A. If the spike level is equal to or greater than the background level of the analyte in the sample, qualify the spiked sample data whenever recovery is outside control limits.

B. If the spike level is less than the background analyte level matrix spike recoveries are not assessed (and therefore not uploaded to LIMS); provide a comment indicating that matrix spike accuracy could not be assessed due to high background analyte levels in the spiked sample. Instead assess the LCS recovery for that analyte and the recovery of other fortified analytes. The LCS recovery and recoveries for other analytes in the sample provides an indication of whether the method was properly applied and was as efficacious as expected. If the LCS recovery for the failing analyte(s) in the matrix spike was outside control limits or if no LCS was prepared, then qualify the associated data as estimates. Also qualify data if the recovery of any other analytes spiked into the sample indicates a systematic bias due to application of the method or matrix effects. If the LCS recovery was within the control limits,

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report associated data as unqualified. If the LCS recovery was outside the control limits the associated data must be qualified as per Appendix 2 or the associated samples reanalyzed for that analyte. It should be noted that, while matrix recovery information may not be available or useful when background levels exceed spike levels, matrix precision derived from duplicate spikes can usually be assessed.

For some analyses (e.g., semi-volatile organics), the accuracy of all individual analytes reported may not be assessed routinely. In those cases, an attempt should be made to group analytes by class (e.g., phenols, PAHs). If any QC check for any compound assessed within a class falls outside control limits, qualify data associated with all compounds in that class. If no class relationship can be established, the group supervisor will use his/her best professional judgment to determine whether to qualify associated data for which there were no matrix QC checks.

Positive and Negative Bias

Laboratory contamination, from reagents and equipment, is sometimes unavoidable, especially in trace level work. Whenever contaminated blanks are detected above the normal method detection limit (MDL), there may be reason to suspect sample results are biased also. When contamination of method blanks is detected, every effort should be made to identify and eliminate the source of contamination. Affected samples associated with preparation batches displaying contamination artifacts above MDLs should be re-analyzed whenever practical or feasible. When samples cannot be re-analyzed or when contamination artifacts cannot be eliminated in method blanks, associated sample results should be qualified as described below.

Measurable contamination artifacts may bias positive sample results detected above the reportable detection limit. Those biases may be especially significant whenever the detected quantity is less than ten times the amount found in the contaminated blank. Therefore, qualify all reported positive results using the 'V' qualifier code when the sample results are less than ten times the amount of contamination detected in the method blank. A comment warning the data user of potential bias should be provided with the qualified data. (This procedure is not applicable to analyses or methods such as 1631 that incorporate a mechanism for handling analytes measured in negative controls.)

Common laboratory contamination artifacts pose a special case when one or more analyte concentrations detected in the method blank approach the MDL. In order for the MDL to achieve a false positive rate of <1%, the EPA-derived MDL must be adjusted to a level assured to exclude 99% of those positive results. This is readily accomplished for analyses that are frequently performed and therefore generate copious amounts of blank data. Historical method blank data collected over recent extended periods (3-6 months) can be used to establish the MDL at the 99^{th} quantile based on statistical analysis of the blank results. It is recommended that a minimum of 50 data points be collected to make statistically robust estimates of the true MDL. Blank data older than one year should be

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used with caution in a non-parametric MDL assessment since older data may not be predictive of current or future false positive rates.

For analyses which do not generate large amounts of blank data, the MDL is set above the EPA-derived MDL using a conservative estimate. As more blank data is accumulated, the MDL can be adjusted as needed.

Other analytical artifacts such as co-elution or interferences may lead to elevated detection limits. In those cases, a comment explaining the reason for reporting elevated detection limits may be appropriate but associated data may not necessarily be qualified. Discretion of experienced analysts and supervisors shall be used in reporting and/or qualifying data when non-contamination derived analytical artifacts are observed.

Laboratory Control Samples (LCS)

The flow chart in Appendix 3 provides step-by-step instructions on addressing LCS failures in multi-component analysis. The 2003 NELAC Accreditation Standard specifically addresses the issue of LCS failure in Chapter 5, Appendix D, Section D.1.1.2. The 2009 TNI Standard (Volume 1, Module 4) specifies that there may be 0 to 5 allowable marginal exceedances of the LCS control limits, depending on the number of analytes in the LCS. Analysts shall monitor the LCS failure rate for individual analytes. Failure of the same analyte in a LCS sample on consecutive analytical runs may indicate a systematic problem and must be investigated. Please refer to Volume 1, Module 4 of the 2009 TNI Standard (Subsection 1.7.4.2) for details.

For data where the number of failures exceeds the number of allowable marginal exceedances or when the same analyte fails on consecutive runs, the following protocol is employed:

LCS Failure Due to a Systematic Error – Analysts must evaluate the analytical system for signs of a systematic problem. If no systematic problem can be detected, the analyst shall note that on the analytical data report or within the comment field (for the LCS) in the LIMS Quality Control Manager (QCM) module. For an established systematic error, the first step is to troubleshoot the method to correct the problem. The data may be reported with qualifiers or reanalyzed, depending on the available options. Consult your supervisor if in doubt on the appropriate actions to take. Even if the data must be qualified, the systematic error must be corrected for future samples. Reanalyze the LCS and associated samples, if possible, once the error has been corrected. If the LCS fails again, then either report the data qualified or start a new analysis cycle, after correcting the problem. Consult your supervisor for instructions prior to qualifying the data.

LCS Failure Due to a Random Error - For random errors, the first step is to simply reanalyze the samples if possible, or to report the data with qualifiers. Consult your supervisor for instructions prior to qualifying the data. If the LCS fails again, then follow the instructions provided above (under the heading for 'LCS Failure Due to a Systematic

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Error). Multiple failures of the LCS may be indicative of a system error and should be more closely investigated.

3.0 ROOT CAUSE ANALYSIS

Root cause analysis (RCA) is a systematic process for identifying "root causes" of problems and an approach for responding to them and adopting measures to prevent the problem from recurring in the future. Quality control failures may be caused by problems that occur at any point in the sample's workflow, from sample preparation to analysis.

QC failures can arise from errors in sample preparation or in the analysis of the prepared sample.

Factors that may contribute to QC failures at the instrument level include:

- Improper instrument maintenance
- Failed or marginal instrument calibrations
- Bad or expired primary or second source standards
- Errors in the preparation of calibration standards
- Mislabeled samples or data entry errors in instrument software inputs
- Errors in sample dilution (at the instrument)

Factors that may contribute to QC failures from sample preparation include:

- Improper calibration of pipettes and balances
- Errors in the preparation of reagents, standards or QC samples
- Contamination of reagents or sample preparation vessels
- Errors in sample dilution prior to preparation (digestion or extraction)
- Mislabeling of samples

3.1 Procedure

When a systemic QC problem or unexpected QC failure is detected, the analyst should begin a systematic enquiry starting with the most proximate potential cause and work their way backwards, eliminating potential causes along the way, until the true cause of the QC failure is identified. The path of the investigation will depend on the nature of the QC failure, the analysis, and the technology employed for the analysis.

For example, low or erratic recoveries for CCVs or LCS samples in ICP-AES might be caused by a dirty or clogged nebulizer, deposits in the spray chamber, unstable plasma or worn peristaltic pump tubing. If a method blank fails high, it could be due to contamination from the autosampler sample tube or from contamination introduced during the preparation of the

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sample. If the blank is repoured into a fresh tube and reanalyzed, with no failure, then the second possibility is eliminated.

3.1.1 Problems at the instrument can usually be diagnosed by checking and comparing calibration standard and second source standard (detector) responses with responses for the same standards from a previous successful run. The analyst should check the instrument maintenance log and the preventive maintenance schedule to make sure that routine maintenance is performed on a regular basis. Usually such maintenance is periodic and conducted on a daily, weekly, or monthly basis depending on the instrument, sample load, manufacturer's recommendations and as documented in the analysis SOP.

3.1.2 At the sample preparation level, the analyst should review records that document the sample's workflow and the metadata generated from support equipment including sample volumes/weights, the calibration logs of pipettes/syringes/balances, temperature records/logs of sample digestion baths/blocks etc. to verify that the preparation SOP was followed faithfully.

For some analyses, especially ones involving time and/or labor intensive sample preparations, a second lab analyst serves as a witness to the primary analyst during critical stages of the preparation of QC samples. Examples include verifying that the correct spike (concentration and volume) is used in the spiking of LCS and matrix spikes.

For solid matrices such as soils, sediments and wastes, it is important that sub-samples of the original sample for matrix duplicates or matrix spike duplicates be as representative of the original sample as possible. Some samples may require additional processing such as particle size reduction by grinding the sample or freeze-drying to remove excess water to enable proper sub-sampling from the original sample.

3.1.3 Once the root cause of a QC failure is identified, corrective actions are implemented and the affected samples reanalyzed if possible. If a cause is identified at the instrument level, the diagnosis and solution to the problem should be documented in the instrument maintenance log. Likewise, for root causes identified in the sample preparation process, suitable corrective actions should be implemented and the affected samples re-prepared and analyzed if possible.

3.2 Documenting Corrective Actions

When the root cause of a QC failure is identified and suitable corrective actions taken, the procedure used to diagnose the problem and implement corrective measures should be documented. The laboratory has a formal system in the LIMS, the Non-conformance Report (NCR) that is used to track and document non-conformances and their resolution (see DEP SOP LB-002). The NCR is linked to specific samples, jobs or events affected by the NCR. If an event has an NCR that affects the reported results, the NCR report for that event is

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included in the final lab report sent to customers. Non-conformance observations that do not affect reported results may also be documented in the NCR system for resolution tracking. The lab's NCR Reporting System is described in SOP LB-002.

3.2.1 When the root cause of a failure is due to instrumental problems, the diagnosis and resolution of the failure should be documented in detail in the instrument maintenance log. If the problem results in data being qualified, then an NCR may be attached to the data.

Common analytical interferences and ways to overcome them are documented in lab analysis SOPs. Not all interferences can be resolved using these standard measures. For example samples composed of uncommon or unconventional matrices may have unresolvable interferences that cause QC failures. In such instances, the sample result is qualified accordingly and accompanied by a comment on the nature of the interference.

3.2.2 Problems that occur at sample preparation that result in data being qualified may be documented in an NCR. Usually the affected samples are reprepared and reanalyzed if sufficient sample is available. If the same or similar problem reoccurs in subsequent analytical runs, it could be an indicator of a systemic problem in the process such as inadequate documentation in the preparation SOP, or inadequate training of sample preparation analysts. Corrective actions in such cases would require a review and possible revision of the SOP by lab supervisors and a review of the initial and ongoing training provided to analysts.

3.2.3 For failures involving Performance Testing (PT) samples, a detailed review of the entire workflow, from sample preparation to analysis is performed. A formal report of the findings and the corrective actions taken is prepared by the lab supervisor in charge of the affected group. The report is then submitted to the lab's QA officer.

Note: *It is strongly recommended that analytical groups save any unused PT sample remaining from a PT study, at least until the final results of the PT study are published. In case of a PT failure, this allows the group to use the remaining PT sample(s) to verify that the corrective actions taken to address the PT failure are successful.*

4.0 REFERENCES

4.1 DEP SOP LB-002, *Non-Conformance Reporting System.*

Appendix of Significant Changes

12/17/2014 Added Section 3 on Root Cause Analysis to the SOP.

11/29/2016 This SOP was edited and checked for 508 compliance.

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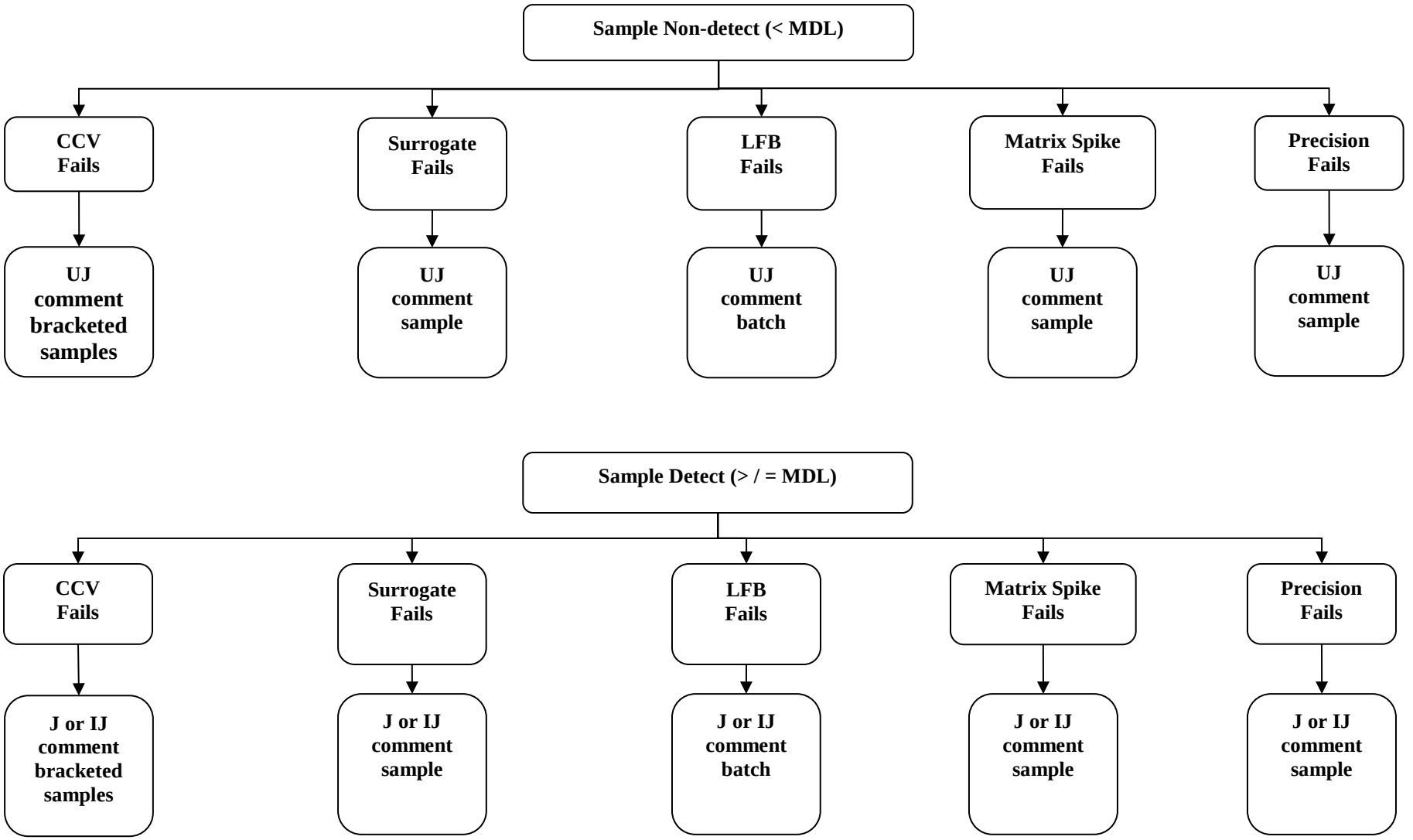
Appendix 1: Data Qualifier Codes

Refer to FAC 62-160 for further details

Code	Description
A	Value reported is the mean of two or more determinations;
B	Result based on colony counts outside the acceptable range; (microbiology)
F	When reporting species, F indicates the female sex;
H	Value is based on field kit determination; Results may not be accurate;
I	Reported value is greater than or equal to the method detection limit and less than the practical quantitation limit;
J	Estimated value; value not accurate; to be used when: » surrogate recovery limits have been exceeded; » no known QC criterion exists; » reported value failed to meet established QC criteria; » sample matrix interference precludes accurate determination; » data is questionable due to improper lab or field protocols; *(Note: a 'J' value shall be accompanied by a justification for its use)* A 'J' value shall not be used if another more appropriate code applies;
K	Off-scale low; Actual value known to be less than the value given;
L	Off-scale high; Actual value known to be greater than the value given;
M	Presence of material is verified but not quantified;
N	Presumptive evidence of material; this qualifier shall be used if: » the compound has been tentatively identified based on mass spectral library search; » there is an indication the compound is present but QC requirements for confirmation were not met;
O	Sampled but analysis lost or not performed;
Q	Sample held beyond the accepted holding time;
T	Value reported is less than the laboratory method detection limit;
U	Indicates the compound was analyzed for but not detected; (*the value associated with the qualifier shall be the laboratory method detection limit);*
V	Indicates the analyte was detected in the sample and method blank;
X	Too few individuals to calculate SCI value
Y	The analysis was from an unpreserved or improperly preserved sample;
Z	Too many colonies were present (TNTC); (microbiology)
?	Data is rejected and should not be used;
*	Not reported due to interferences;

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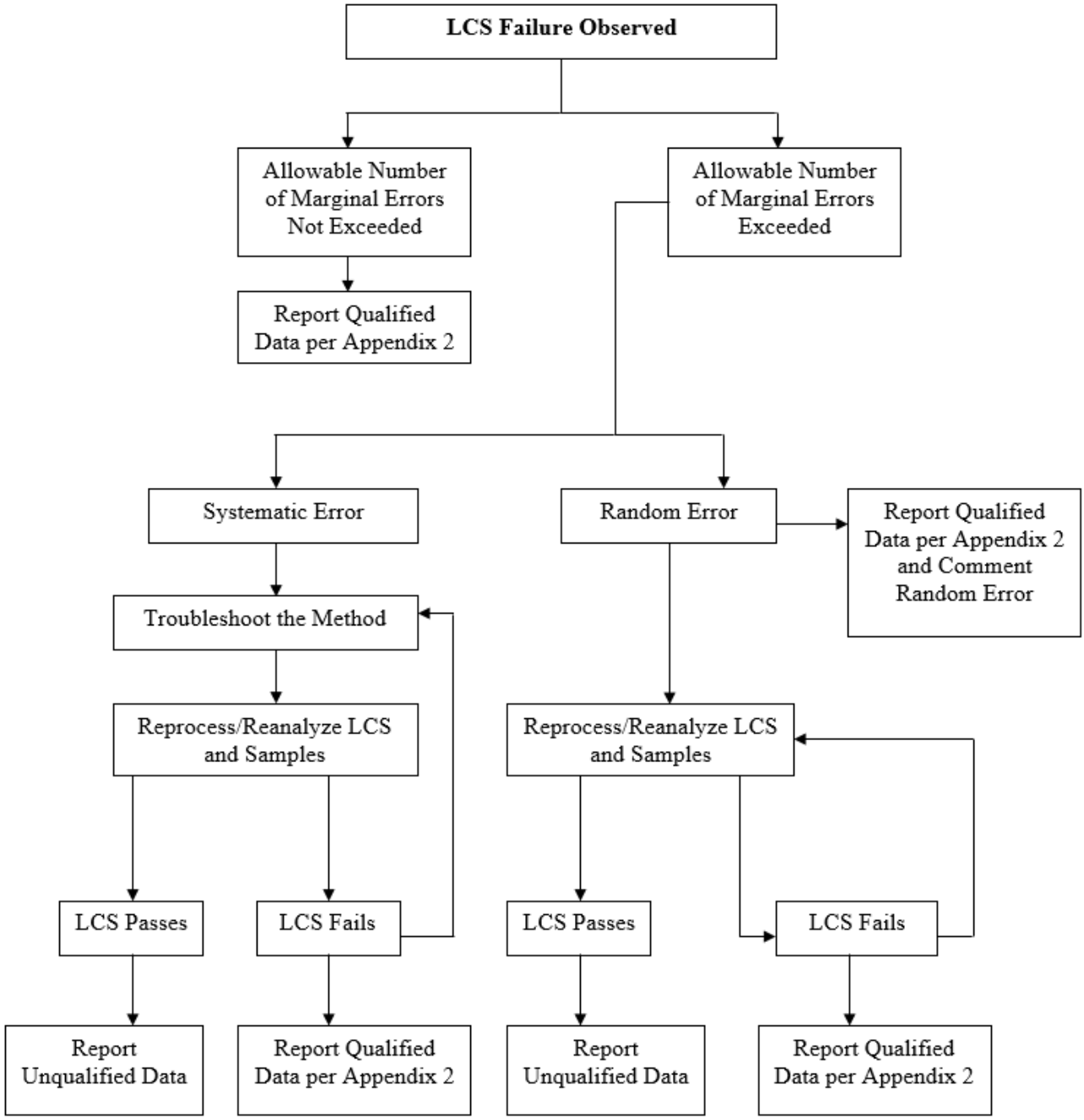
Appendix 2: Strategy for Qualifying Data Associated with Quality Control Failures



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Appendix 3: NELAC 2003 LCS Failure Protocol

Section D1.1.1.2



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