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Approved by: [signature] Date: 2-9-2018

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CONTROLLED DOCUMENT

Purpose

The purpose of the laboratory quality audit is to systemically evaluate ongoing laboratory operations in order to assure that the analytical data produced by the laboratory meet the quality criteria established for their intended use. The audit is a tool to be used to assess the effectiveness of the quality program; to identify areas requiring improvement; and to be used in training analytical staff in the requirements for sound quality policy.

Because the Office of the Texas State Chemist is a regulatory agency whose mandate is to enforce the Texas Commercial Feed and Fertilizer Control Rules it is imperative that the data generated by the Agricultural Analytical Services branch of the Office be of the highest quality in terms of defensibility. Routine audits help assure that the data generated by the laboratory are defensible, i.e., that they meet prescribed standards for precision, accuracy, traceability, representativeness, completeness and comparability.

The attached checklist covers the major aspects of laboratory operations. It is in most respects a generic document, but it has been tailored to the operations of the Office of the Texas State Chemist and to our way of performing and documenting analyses. As such, it can and will evolve as needed to accommodate system and procedural changes.

Scope / Field of Application

This will be a process or vertical audit that will focus on a particular method and will audit all the inputs, operations, and activities required to produce the result.

This is a document designed to be used as an aid during a physical audit of the analysis of a single analyte. The scope of the audit is not so limited however. Many laboratory support functions (e.g., routine equipment calibration/maintenance, training, standard operating procedures, validation, etc.) are also addressed while performing the audit. An effective quality control program is comprised of an integrated collection of activities and the audit scrutinizes and reevaluates each activity on a regular basis.

Responsibilities

Responsible chemist (or analyst): the analyst(s) who actually performed the analysis on the audited analyte and is responsible for following the quality system procedures.

Current chemist (or analyst): the analyst(s) who is currently assigned to perform the audited analysis; may or may not be the responsible chemist.

Reviewing chemist: the analyst or manager who does the final document review to make sure that all paperwork supporting a given analysis is complete.

Auditor: A manager or analyst who has been trained as an Internal Auditor and has the background and/or experience in the method.

Quality Manager: responsible for developing audit procedures, establishing schedules, ensuring auditors are competent for the activity, and maintaining audit records.

Procedure

Some terminology used in the Audit Checklist:

§ **Volumetric solutions (Standard Solutions):** solutions of reagents of known concentration intended primarily for use in quantitative determination.

§ **Test solution:** solutions of reagents of approximate concentration that are typically used for qualitative determinations or are used "in large excess" in a quantitative analysis.

§ **Bulk reagent:** any reagent in its original delivery container.

CHECK LIST:

Sample Analysis Audit Checklist	
Date audit was performed:	
Auditor:	
OTSC Sample Number:	
Analyte:	
Date(s) analyzed:	
Responsible Analyst:	
Current Analyst:	
Reviewing Analyst:	
Current Manager:	
Responsible Manager:	

In preparing for the audit, the auditor should carefully read the appropriate quality system procedures and the Standard Operating Procedure for the audited analyte. Having a list of details to be reviewed (reagents, equipment, standards, times, temperatures, etc.) helps focus the audit.

List all documentation reviewed in preparing for and conducting this audit (check box next to item if applicable):

A. ☐ SOP's: AAS Method _____, AOAC Method _____, M0021, M0028

B. ☐ Documentation of worksheet ID _____
Analytical Summary Sheet (Report Coversheet)
Worksheet Reports

C. ☐ Lab Notebook #(s) _____

D. ☐ Balance calibration records

E. ☐ LIMS database for worksheet _____

F. ☐ Working Control database; filename _____

G. ☐ Pipette Calibration Records _____

H ☐ Oven/Incubator/Waterbath/Freezer/Refrigerator Log(s)

I. ☐ ICN database _____

J. ☐ Method Validation, Training Records_____

K. ☐ Sample receipt, preparation _____

L. ☐ FFCS database reported results_____

M. ☐ Other: _____

N. ☐ Other: _____

Generally speaking, if the answer to an audit question is No, and if there is a space for "Comments" then an explanation of the deficiency is required.

DOCUMENTATION

Is there a most-recently approved copy of the analytical standard operating procedure for the audited analyte available in the appropriate file for the analyst to refer to during the analysis?

☐ No ☐ Yes ☐ Not applicable

Comments:

Is the standard operating procedure consistent with the compendial method it purports to represents? (Non-trivial deviations from the official method must be validated before implementation.)

☐ No ☐ Yes ☐ Not applicable

Comments:

Are all data and supporting documents immediately available and easily retrievable? (All documentation relating to a particular analysis should be able to be retrieved within 4 hours. Required data includes, but is not limited to, Analytical Summary Sheet (Report Coversheet), worksheet reports , lab notebook with solution preparation information, refrigerator/chiller/oven temperature logs, equipment maintenance and calibration logs, computer and instrument output, control charts, etc.)

☐ No ☐ Yes ☐ Not applicable

Comments:

Does the documentation contain sufficient information and explanation such that the analysis report can be readily interpreted by knowledgeable persons other than those responsible for their generation?

☐ No ☐ Yes ☐ Not applicable

Comments:

Does the documentation contain sufficient information such as would allow, when desirable, satisfactory repetition of the analysis under the original conditions?

☐ No ☐ Yes ☐ Not applicable

Comments:

Are all original entries contained in the documentation neat and legible and recorded using black or blue, permanent, water-resistant ink?

☐ No ☐ Yes ☐ Not applicable

Comments:

If the procedure used to analyze the sample is not a compendial procedure, was the procedure validated?

☐ No ☐ Yes ☐ Not applicable

Comments:

If the procedure used to analyze the sample is a validated procedure, is the validation documentation archived and readily available?

☐ No ☐ Yes ☐ Not applicable

Comments:

If there are any corrections or changes to any entries in the documentation, were they properly made? Proper corrections are made as follows:

§ No erasures, correction fluid or correction tape may be used while making corrections to any documents.

§ Draw a single line through the incorrect entry; **do not obliterate the original entry—it should be legible through the strikeout.**

§ Write the correction in as close a proximity to the original as possible.

§ The analyst's initials should appear near the correction.

§ The date of the correction should appear near the correction (unless a date is being corrected, in which case the correction dates itself).

§ **If the reason for the correction is not obvious,** then there should be a brief explanation of why the change was necessary. The explanation should be written near the correction if possible, otherwise it can be referenced elsewhere on the page or attached sheet using an "*".

☐ No ☐ Yes ☐ Not applicable

Comments:

Does the Analytical Summary Sheet (Report Coversheet) provide for recording the residence times, temperatures, digestion times, bath temperatures, etc. for all **critical** steps defined in the standard operating procedure and identify the responsible person?

☐ No ☐ Yes ☐ Not applicable

Comments:

If appropriate, are special storage or handling requirements indicated on the Analytical Summary Sheet (Report Coversheet)?

☐ No ☐ Yes ☐ Not applicable

Comments:

Are current samples requiring the same analysis being properly stored/handled?

☐ No ☐ Yes ☐ Not applicable

Comments:

Are all weights below 1 gram in the form 0.xxxx (including preceding zero)?

☐ No ☐ Yes ☐ Not applicable

Comments:

If inserts are used (i.e., paper such as chromatograms, printouts, etc.) are they permanently attached (stapled, taped, copied) or placed in a separate envelop (fully cross-referenced to the original set and easily retrievable)?

☐ No ☐ Yes ☐ Not applicable

Comments:

During the analysis was any instrument output generated? Are all entries on instrument output properly labeled (e.g., concentrations of calibration standards, ID of working standards, individual sample numbers for investigative samples, sample dilutions [if different than described in the standard operating procedure], etc.).

☐ No ☐ Yes ☐ Not applicable

Comments:

Is the computer database information consistent with paper documentation?

☐ No ☐ Yes ☐ Not applicable

Comments:

If any results were generated using external software (i.e., using, for example, a spreadsheet program), was the external program validated before use, and is the validation documentation archived and readily available?

☐ No ☐ Yes ☐ Not applicable

Comments:

Are any comments, if given, reasonable, complete, and supportive of data observations within the set documentation?

☐ No ☐ Yes ☐ Not applicable

Comments:

Was the set properly reviewed by the reviewer as described in SOP M0021?

☐ No ☐ Yes ☐ Not applicable

Comments:

Are all required blanks either filled in or correctly marked through?

☐ No ☐ Yes ☐ Not applicable

Comments:

Is each page of the documentation consecutively numbered in the format "m/n" or "m of n", where m is equal to the consecutive page number, 1,2,3,…,n, and n is equal to the total number of pages? (Self-paginated spreadsheet and instrument printouts may be considered as one page).

☐ No ☐ Yes ☐ Not applicable

Comments:

Is the worksheet/set number included on all required pages of the documentation?

☐ No ☐ Yes ☐ Not applicable

Comments:

Is each required page of the documentation signed and dated by the responsible analyst? Only the last page of the worksheet report needs to be signed and dated. Only the last page of spreadsheets and instrument printouts need to be signed and dated.

☐ No ☐ Yes ☐ Not applicable

Comments:

REAGENTS AND SOLUTION PREPARATION

Is there an up-to-date Approved Vendor's list?

☐ No ☐ Yes ☐ Not applicable

Comments:

Were all volumetric and test solutions used before their respective expiration dates?

☐ No ☐ Yes ☐ Not applicable

Comments:

Are unique identification numbers for all bulk reagents, volumetric solutions, and test solutions used during the course of the analysis included on the Analytical Summary Sheet (Report Coversheet)?

☐ No ☐ Yes ☐ Not applicable

Comments:

. Are all unique identification numbers for bulk reagents, volumetric solutions, and test solutions in the required format and recorded in the laboratory notebook?

☐ No ☐ Yes ☐ Not applicable

Comments:

Were all bulk reagents used in the preparation of solutions for the audited analysis used before their respective expiration dates?

☐ No ☐ Yes ☐ Not applicable

Comments:

Are the preparation dates in the laboratory notebook for each volumetric and test solution consistent with the dates of use indicated on the Analytical Summary Sheet (Report Coversheet)?

☐ No ☐ Yes ☐ Not applicable

Comments:

Are the unique identification numbers on the **current** volumetric and test solutions consistent with the preparations described in the laboratory notebook?

☐ No ☐ Yes ☐ Not applicable

Comments:

Are all samples, extracts, reagents, reference materials and standards stored so as to preserve their identity, concentration, purity and stability?

☐ No ☐ Yes ☐ Not applicable

Comments:

Are all calibration standard or reference standard materials used in this analysis traceable to a (primary) Certified Reference Material such as NIST or USP?

☐ No ☐ Yes ☐ Not applicable

Comments:

Does the ICN Database information agree with the manufacturer's label on any bulk reagent listed?

☐ No ☐ Yes ☐ Not applicable

Comments:

LABORATORY NOTEBOOKS

Is each separate reagent or solution preparation on a given page of the laboratory notebook initialed and dated by the analyst, and does the description include the following?

§ A unique identification number for each prepared solution.

§ The name/concentration of the volumetric or test solution being prepared (e.g., 0.15 *N* HCl in methanol).

§ The name (and molecular formula if more than one form is possible) of the bulk reagent(s) being used in the preparation of the volumetric or test solutions.

§ The ICN assigned from the database for each bulk reagent.

§ Weights [(gross, tare, net) or (weight by difference)] or volumes, expressed in appropriate units, of the bulk reagent used in the preparation of the volumetric or test solution.

§ The sequence of dilutions (if necessary) used to prepare the final concentration of the volumetric or test solution, including volumes taken and final volumes.

§ The unique identification of the balance or pipettor used.

§ Expiration date of the prepared solution.

§ Identification of the water source.

☐ No ☐ Yes ☐ Not applicable

Comments:

INSTRUMENTATION AND EQUIPMENT

Does the Analytical Summary Sheet (Report Coversheet) provide an entry to record the unique ID number of each significant piece of equipment that was used in the analysis? (i.e., balance, oven, incubator, pipettor, thermometer, titrator, instrument, etc.)?

☐ No ☐ Yes ☐ Not applicable

Comments:

Of the listed equipment requiring calibration or maintenance, was each properly performed?

☐ No ☐ Yes ☐ Not applicable

Comments:

Is the balance calibration check for the balance used in the audited analysis documented for the week of the analysis? Is the balance in-control?

☐ No ☐ Yes ☐ Not applicable

Comments:

Are any temperature recordings out-of-control for any listed equipment requiring day of use monitoring?

☐ No ☐ Yes ☐ Not applicable

Comments:

CONTROL CHARTS

Are the working control data for the set located on the Analytical Summary Sheet (Report Coversheet), and is this data in-control for the audited analyte? If there is a trend in the previous year's working control data, has it been addressed in the control chart comment section?

☐ No ☐ Yes ☐ Not applicable

Comments:

Is the control chart database up-to-date for the analyte being audited?

☐ No ☐ Yes ☐ Not applicable

Comments:

Have the control chart subgroup statistics been correctly calculated? Is it documented and filed properly?

☐ No ☐ Yes ☐ Not applicable

Comments:

VI. MISCELLANEOUS

Are any deviations from the protocol apparent from either the audited documentation or from interviewing the analyst(s)? Explain.

☐ No ☐ Yes ☐ Not applicable

Comments:

If there were any deviations from the protocol, were they approved by the supervisor or a lab manager and explicitly documented?

☐ No ☐ Yes ☐ Not applicable

Comments:

What was the Z-value on the last check sample. Was the corrective action procedure followed if z >3?

☐ No ☐ Yes ☐ Not applicable

Comments:

Is there a trend in the previous year's check sample control charts?

☐ No ☐ Yes ☐ Not applicable

Comments:

Revision History

August 2012, New format.

August 2013, Edited to specify audit will be completed during the first quarter of the calendar year.

October 2014, Edited to clarify that the audit should include the management elements of ISO 17025 in addition to the technical or testing elements.

July 2016, SMW Changed title and scope of audit to limit use to technical requirements for specific analyte/method (Created M0026 for more inclusive system audits.) Removed edit from August 2013, specifying audit completion during first quarter. Removed safety related section. Minor edits were made to reconcile checklist with ISO 17025 and Internal Audit training.

February 2018, MKR; Revision number will stay at 4 because revision was never released when July 2016 revision was done. Additional formatting changes to document. General review.