



SOP ON ASSURING THE QUALITY OF ANALYTICAL RESULTS

Compiler: O.Mwaniki

Signature.....

Approver: P. Kamuti

Signature.....

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SOP ON ASSURING THE QUALITY OF ANALYTICAL RESULTS

1. Scope

This document describes the quality control procedures for monitoring the validity of test results undertaken within the laboratory, as well as ensuring that data is transferred accurately.

2. Procedure

- a) The laboratory shall monitor analytical performance through operating systematic internal quality control checks in such a way that trends are detectable.
- b) All results obtained shall be documented, recorded and used for quality assurance programs.
- c) The laboratory shall implement the use of reporting templates to avoid errors in data transfer and entry and provide for better traceability of data.

A. Internal Quality Control:

i. Linearity

Linearity is the exponential proportionality of the response of the machine with change in concentration. The correlation factor should be greater than 0.95 for perfect linearity. The interpretation of the results should always be within the calibration curve. Linear regression shall be used when making calibration points in any batch.

ii. Accuracy of test methods:

a. Participation in proficiency test.

The test methods used by laboratory shall be monitored for accuracy by use of proficiency test results. The laboratory shall only participate in proficiency tests from accredited proficiency testing providers.



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The PT providers shall be accredited according to ISO 17043. PT results shall be acceptable if within ± 2 standard deviation for all analytes within laboratory scope. The results are computed by use of the z-score.

The z-score computation shall be evaluated using the formula
$$z = \frac{x - x_a}{\sigma_p}$$

Where:

- x is the mean of the laboratory results
- x_a is the median value of the inter-laboratory data (or proficiency testing data) or assigned value.
- σ_p is the standard deviation of all the results / standard deviation for proficiency assessment.

NOTE: The standard deviation used is the standard deviation of the mean (standard error of the mean, s.e.m), given as:

$$s.e.m = \frac{\sigma}{\sqrt{n}}$$

The z-score shall be used in the ACL laboratory to measure accuracy of inter-laboratory results (organised by this laboratory).

All PTs, CRM and internal standards are discussed and any non-complying results entered in non-conformance form. These results i.e. PT results and corrective actions are also discussed in the management review meetings

b. Certified reference materials

The laboratory shall use reference materials where possible as a measure of accuracy. Where a certified reference material is not available, internal standards shall be used to assure quality. The acceptance criteria for z-scores for all the analytes tested when using certified reference material shall be ± 2 .

c. Recovery

The test method used by the laboratory shall be checked for recovery at LOQ for every batch whenever possible.



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The % recovery shall meet method requirements before samples are reported to the client.

B. Evaluation of Training and Monitoring Competence of Analysts

Competence test is a useful measure for the following;

- 1) Establish the competence of the analyst.
- 2) Know the accuracy of the method.
- 3) Determine the level of confidence of the results.
- 4) Performance and sensitivity of the machine i.e. proof of linearity and the limit of detection and Quantitation of the machine.

C. Repeat analysis of inorganic analyses and pesticide residue analysis is necessary when:

- i. The correlation, r^2 is less than 0.95. This shows that the detector is not approaching perfect linearity as described in competency evaluation.
- ii. The percentage recovery in pesticide residue or total carbon analysis is outside the set limits by the control charts, and a further re-calculation of the twenty previous percentage recoveries and total carbon in soil analysis is still outside the limits in the recalculated quality control chart.
- iii. The measure of trueness evaluated as:
 - (a) Precision evaluated with F test shows that $F_{calc} > F_{critical}$. This indicates that the data has no precision.
 - (b) Accuracy evaluated with t test shows that $t_{calc} = \pm t_{critical}$. This shows that data evaluated is not accurate.
- iv. The repeat analysis shall be repeated after authorisation from the Technical Officer or Quality Officer or the assistant or the Sample supervisors.



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D. Transfer and Checking of Data:

- i. After completion of all analysis (repeat or otherwise) data shall be recorded on the worksheet and entered into the report template for the specific analysis carried out (e.g. for analysis of soil for fertility evaluation, the results shall be entered in the QS template)
- ii. The analyst shall then print the report template and present to the sample supervisor or the person acting on behalf of the supervisor to check the data and data transfer.
- iii. The respective sample supervisors/ analyst confirm systematic transfer and calculations are accurately done, check whether results are entered on j score and meets the criteria as set by the j score of respective test method
- iv. Prepare and sign the draft report.
- v. The sample supervisor shall hand over the draft report, filled work sheet to secretary Head ACL and food safety (Deputy Director in charge of the lab) who shall then prepare the final report.
- vi. The analyst participating in analysis, technical signatory and Head ACL and food safety laboratory sign the verified final report.
- vii. The final results are stored in the Head ACL and food safety office before dispatched to the client.

E. Monitoring the Quality of Test Results

i. Pesticide residue analysis:

- a) The laboratory shall use J score charts for trends analysis.
- b) J-score for malathion-D10, Dimethoate-D6 responses at 25 and 50 mg/kg shall be used as procedural and equipment internal standards for LCMSMS while Dichlorvos- D6 and Gamma HCH-D6 at 50mg/kg shall be used shall be used as procedural and equipment internal standards for GCMSMS analysis.
- c) If the results in part b falls between average and $\pm 30\%$ then the results are reported to the client. If any of the internals standards for both GCMSMS or



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LCMSMS falls outside $\pm 30\%$ then the results are not reported to the client and investigation initiated by technical signatory.

ii. Inorganic methods and Soil analysis:

- a) The method acceptability criterion for inorganic analysis shall be evaluated by entering the result values for the reference material or internal standards in the J- score chart. If 4 consecutive results for any of sample-A fall between ± 1 SD and ± 2 SD, then investigation should be done before results are released to the client. If two consecutive results of sample-A fall outside ± 2 SD, then investigation should be done before results are released to the client.
- b) The average correlation should not be less than 0.95 of all values.

iii. Formulation analysis:

The laboratory shall participate in proficiency testing at least once a year and the results obtained used to compute accuracy and uncertainty. For routine quality control, the data from the results shall be plotted in the J score charts. If 4 consecutive results of the difference between two replicates fall between ± 1 SD and ± 2 SD, then investigation should be done before results are released to the client. If two consecutive results of the difference between two replicates fall outside ± 2 SD, then investigation should be done before results are released to the client.

iv. Aflatoxin analysis

The laboratory shall participate in proficiency testing at least once a year and the results obtained used to compute accuracy and uncertainty.

a) System suitability

Aflatoxins mix of 9.0 ppb (B1, B2, and G1 & G2) are injected 3 times. Calculate the % RSD of peak area and retention time. The acceptance criteria of %RSD of peak area and retention time are less than 20.



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b) Calibration curve

The matrix standard curve of AF B1, B2, and G1 & G2 is prepared at the concentration of 1.0, 3.0, 9.0, 30.0, 50.0 and 100 ppb in blank matrix. The Acceptance Criteria of $R^2 \geq 0.95$

c) Spiked recovery

Spike 10 g of Blank sample with 200 μ l of 1mg/l aflatoxin mix working solution.

The amount of aflatoxin mix standard added to the sample blank theoretically corresponds to 0.005 μ g/kg of Aflatoxin B1, B2, G1, G2 & Ochratoxin A. Fumonisin is spiked at 0.05 μ g/kg

The acceptance criteria of % recovery should be in the range of 70 – 120% for B1, B2, G1, G2 following SANTE/11945/2017 Rev. Annex II regulation.

$$\% \text{ Recovery} = \frac{(\text{Conc. in spike} - \text{conc. in blank})}{\text{Actual Spike conc.}}$$

d) Analyses Blank

There is a blank sample per batch. The injected analyses blank has S/N ratio of less than 3.

e) Confirmation

Retention time: The positive sample should have the retention time in the range ± 0.2 min compared to standard solution.

Ion ratio of precursor ion and daughter ion:

The positive sample with aflatoxin (B1, B2, G1 & G2), Fumonisin (B1&B2) Ochratoxin A should have the ion ratio in the range $\pm 25\%$ compared to the ion ratio of standard.

F. Analysis and Review of Monitoring Data (J-Score Charts)

a) Suitable control limits for the analytical determinations shall be calculated to ensure that trends in analysis are detectable.



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- b) The data obtained from analysis of reference materials or internal standards and any other measures of control (e.g. recovery data) shall be plotted on control charts derived e.g. J-Score templates.**
- c) The control chart results shall be discussed quarterly to ensure that the trends in analysis are detected and under control. This shall be done in meetings with analyst responsible analysis or during technical meetings.**

G. Out of Control Investigations

In case of deviation from any of the pre-defined quality controls, the analyst who performed the test will inform the technical team leaders who will initiate root cause analysis and inform the quality assurance officer.



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3. Amendment List

Amendment No.	Date approved	Nature of amendment
1	11/04/07	Deletion of use of repeat samples

4. Revision List

Revision No.	Effective date	Nature of revision
1	11/04/07	As per the amendment.
2		Inclusion of the criterion for calculation of data – linearity, accuracy, limits of detection and quantification.
	18/06/08	Inclusion of criterion for monitoring of analytical results.
3		Inclusion of criterion for monitoring of analytical results.
	02/10/09	Inclusion of the calculation of precision, accuracy and uncertainty for formulation analysis.
4		Change of the r^2 acceptance value from 0.99 to 0.95
5	21/2/11	Inclusion of reference to SOP MO3023 on the determination of expanded uncertainty.
	26/4/11	Change of the trend limit for analysis in the quality control chart (i.e. between upper +2 and +3 standard deviations or -2 and -3 standard deviations) to 0.95 from 0.9
6		Inclusion of the use of Control charts, 5(iii).
7	13/5/11	Inclusion of linearity acceptance criterion for formulation analysis.
	08/05/15	Inclusion of calculation of accuracy using Use of the t-test and the computation of z-Score (Section D. 1 and 2)
8		Changed compiler and approver
		Include header of the document
9	22/06/15	Interchanged If F_{calc} is $< F_{critical}$ with If F_{cal} is $> F_{critical}$ in part 3c
		Included the acceptance criteria of z-scores



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10		Included statement "The septum and liner shall be changed after every batch of 18 samples. The column shall be trimmed on the injector side once every two weeks and changed after every six month and conditioned appropriately. The LC columns shall be changed every six months" in part 5 i b c
11	5/12/2017	
12	30/10/18	Introduced "The laboratory shall use J score charts for trends analysis. For GC-ECD analysis, Lindane (early eluting), Alpha cypermethrin (mid eluting), and Deltamethrin (late eluting) shall be injected at 0.05mg/kg and their responses recorded in the J score charts. If the chart signals "investigation necessary" the analysis should be stopped awaiting investigation and maintenance which may include but not limited to change of septum, liners and trimming of column." and "The results of recoveries for the molecules mentioned in (a) above shall be entered in the J score chart to ensure the method is in optimal performance. If 4 consecutive results for any of these molecules fall between ± 1 SD and ± 2 SD, then investigation should be done before results are released to the client. If two consecutive results of any of the above molecules fall outside ± 2 SD, then investigation should be done before results are released to the client.
13	08/02/2021	In part 5 (i) Introduced "The method acceptability criterion for inorganic analysis shall be evaluated by entering the result values for the secondary reference material (sample A) in the J score chart. If 4 consecutive results for any of sample-A fall between ± 1 SD and ± 2 SD, then investigation should be done before results are released to the client. If two consecutive results of sample A fall outside ± 2 SD, then investigation should be done before results are released to the client" and "The average correlation should not be less than 0.95 of all values" in part 5 (ii)
14	06/05/2021	Inclusion of statement "For routine quality control, the data from the results shall be plotted in the J score charts. If 4 consecutive results of the difference between two replicates fall between ± 1 SD and ± 2 SD, then investigation should be done before results are released to the client. If two consecutive results of the difference between two replicates fall outside ± 2 SD, then investigation should be done



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		before results are released to the client.
15	15/12/2021	In part 5 (iii) included "The control chart results shall be discussed periodically to ensure that the trends in analysis are detected and under control.
16	25/01/22	Part 5 (iii) reviewed to read; "The control chart results shall be discussed quarterly to ensure that the trends in analysis are detected and under control. In part 3. A (iii) where reference standard (s) is unavailable, the laboratory shall use CRM's or retained PT material as an alternative. Revision to include requirements of ISO 17025-2017 version, Format the table of contents. Inclusion of mycotoxin quality control procedure part 2.4 in the SOP
17	03/07/2024	Inclusion of a responsible person to initiate investigations in part 2(f) of the SOP G0512 when reference material results fall out of control. Formatted the procedure to include Deputy Director of laboratory and number

5. References

- Statistics for Analytical Chemistry, Fifth Edition, by J. C. Miller and J. N. Miller.
- ISO 17025-2017.

6. Records

- Laboratory workbooks
- Laboratory worksheets
- Chromatograms
- Equipment data printouts
- Equipment log registers.