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Purpose

The proficiency testing plan properly establishes the procedures required to implement the PT program and to meet the ISO/IEC 17043:2010 standard 4.4.1. The reference material production plan establishes the procedures required to produce reference material that meet standards set forth in ISO 17034:2016. Accompanying certificates produced will meet the requirements of ISO Guide 31.

Scope / Field of Application

This procedure applies to the PT program administrated for APTECA and FAO. The procedure also covers production of items deemed suitable for use as reference materials.

Responsibilities

State Chemist: Make executive decisions with regard to the PT/RM program implementation.

Quality Assurance Manager: Ensure the PT/RM program follows the ISO/IEC 17043 and ISO 17034 standards.

RM Coordinator: Coordinate each step in the program, oversee the reference material production, storage, and PT/RM distribution

IT Support Staff: Website maintenance, generation of PT testing reports.

Procedure

PT Program Plan

a) Proficiency testing provider name and address: Office of the Texas State Chemist, 445 Agronomy Road, College Station, TX USA 77845

b) Coordinator information: Refer to the website of HTTP://APTECA.TAMU.EDU

Subcontractors: NA

c) Participation criteria: A testing party that is currently involved in aflatoxin testing globally.

d) Participation in the PT program is currently limited to 500 or less participants based on the current program capacity. The participating laboratories include but are not limited to: Industry aflatoxin testing laboratories, government testing laboratories, and laboratories from academic institutions. Laboratory of specialty can be considered in a case by case situation.

e) The program is to measure mycotoxin in cereal and oil seeds.

f) The measurement of aflatoxin concentration ranges from 0 µg/kg up to1000 µg/kg.

g) Potential source of error: uncertainty associated with the reference material production;

h) Requirements for the production, quality control, and storage of the proficiency test items: The production of the PT test item shall follow SOP M0053.

i) Requirements for distribution of the proficiency testing item: Item distribution will utilize the following mechanisms: Distribution through mail and package carrier such as Fedex, DHL, UPS, or USPS.

j) Collusion among the participants and/or falsification of results are prohibited. All participants need to follow the PT instructions and report their test results based on said instructions. Any falsification of the testing results would initiate an investigation. The immediate statistical analysis will exclude the falsified result. In addition, participant's future eligibility in the PT program will be evaluated.

k) The PT program information will be made available to the participants through the website at: HTTP://APTECA.TAMU.EDU. The instructions include: time schedule of the PT, information on analyzing the sample, and results reporting. The shipped PT item will be ready for analysis, and further regrind will not be necessary. When analyzing and reporting the sample, a separate portion of the sample will be weighed for each complete analysis. Analysis will be performed as it is usually performed by the laboratory. The analytical results will be reported in the spaces provided on the report form or directly by using the result reporting portal on the website: HTTP://APTECA.TAMU.EDU . The individual analysis results will be reported and not averaged into a single value.

l) The PT program does not require a specific method to be used for the PT item testing. The PT program shall offer data evaluation for different testing methods,

when applicable.

m) The homogeneity and stability testing of the PT item should follow SOP M0053. The stability test shall be performed at the inception of one unique matrix and/or unique analyte. Once the stability of certain matrices has been established, the stability test can then be performed on an "as needed" basis.

n) The website reporting portal will use a credential based log in system. The report requires the laboratory to use the online form. The online form requires two entries per PT item from the participating laboratory. The concentration of the web input for aflatoxin is in µg/kg.

o) The statistical method used to analyze the reported data shall use average, standard deviation and relative standard deviations. The outlier test is based on the Cochran test and Grubbs test.

p) The assigned value of the PT item shall be determined by averaging the homogeneity testing results by OTSC. The concentration of the PT test item shall be reported in µg/kg. The measurement uncertainty of the assigned value is the standard deviation divided by the square root of the number of measurements. The measurement uncertainty of the individual results from the OTSC is the Relative Standard Deviation (of the multiple homogeneity test measurement results) multiplied by 2. The assigned value shall not be disclosed to the participants ahead of the data report and evaluation.

q) The evaluation of the participants will be through the application of the z score calculation. The z score shall be calculated based on the following equation:

$$z = (x - \mu) / \sigma$$

where z is the z score to evaluate the individual laboratory performance, X is the mean value of the two individual laboratory reported number, and µ is the assigned value for the corresponding assigned value. σ is calculated based on the following equation:

$$\sigma = \{Cx2^{[1 - \log(C)/2]}\}/100$$

where C is the concentration of aflatoxin.

Z score evaluation:

$|z| \leq 2.0$ satisfactory

$2.0 < |z| < 3.0$ questionable

$|z| \geq 3.0$ not satisfactory

r) The final report to each individual participant will include the testing results from each individual laboratory with the laboratory name removed and replaced with numeric numbers. The Z score of the participating laboratory shall be reported. The data will be reported back to the participants on the webpage with the credential

login.

s) The PT program treats failure of on time data submission as abandonment of program participation. If a PT item is lost or damaged during the testing and reporting round: 1) a new PT item will be shipped out to the laboratory immediately upon the notification of PT item loss or damage. The program treats no report of loss or damage as a recognition of successful sample delivery. 2) If the lab receives the original shipment or the replaced shipment and is not able to return the testing results in a timely manner as required for the PT program, the lab will be excluded from the final statistical analysis. If a PT item is prohibited from shipping to the enrolled laboratory in countries that have shipping restrictions, the participant laboratory will then be removed from the PT program and will not be included in the evaluation.

PT Item and Reference Material Production and Inventory Control Plan

a) The storage condition of the reference material shall be closely monitored for temperature variations.

b) The production coordinator shall identify appropriate material for reference material production and assess the commutability of the reference material as produced. For aflatoxin concentration determination, independent analyses by methods other than the primary HPLC methods can be performed when necessary and appropriate.

c) The quality manager shall issue certificates and other related documentation for the PT item or the reference material accordingly.

d) PT samples will be distributed in heat-sealed packets of an appropriate weight. A label will be securely affixed to each packet identifying the PT program round and the RM batch # from which it was derived.

e) RM samples will be packaged in 1L bottles containing approximately 500g. The lids will be securely tightened. A label will be securely affixed to each bottle identifying the material, the RM program, and the batch #. Each bottle in a batch will also be given a sequential number. The label will be designed to remain legible and intact under the defined storage and handling conditions throughout the lifetime of the RM.

f) The quality manager shall ensure that the reference material and PT item labelling and packing comply with safety regulations. Accordingly, the shipping arrangement shall comply with shipping regulations.

g) The quality manager shall take appropriate measures to ensure the integrity of the PT items and reference material. Periodic audits should be performed to check PT item and reference material integrity.

Data Management Plan

a) The software for data processing (i.e. homogeneity test, stability test) will be validated by two qualified personnel, which might include a statistician. The computer and data storage system needs to be backed up on a fixed schedule. System recovery shall follow the IT department policy.

b) Results from PT participants, after submitted through the on-line portal, shall be recorded and stored on the designated computer server. A two-step validation shall be implemented as follows:

   a. The on-line calculation tool shall be validated by at least two qualified personnel, which might include a statistician.

   b. The calculation for each PT round will be processed by the on-line calculation tool. At least 5 % of the final calculated results will be manually validated by a qualified person.

c) Results that are inappropriate for statistical evaluation: The outlier test will be using Cochran C test and Grubbs test (Appendix A and B). The calculation will be based on the following equation:

$$C_j = \frac{s_j^2}{\sum_{i=1}^{N} s_i^2}$$

Where

C_j = Cochran's C statistic for data series j

s_j = standard deviation of data series j

N = number of data series that remain in the data set; N is decreased in steps of 1 upon each iteration of the C test

s_i = standard deviation of data series i ($1 \leq i \leq N$)

d) The Grubbs test statistic is calculated based on the following equation:

$$G = \frac{\underset{i=1,\ldots,N}{max} \left| Y_i - \bar{Y} \right|}{s}$$

Where $\bar{Y}$ and s denoting the sample mean and standard deviation, respectively.

References

1. ISO/IEC 17043:2010 Conformity assessment-General requirements for proficiency testing.
2. ISO Guide 34: 2009(E) General requirements for the competence of reference material producers.

3. ISO/IEC 13528:2015 Statistical methods for use in proficiency testing by interlaboratory comparison.
4. W.G. Cochran, The distribution of the largest of a set of estimated variances as a fraction of their total, Annals of Human Genetics (London) 11(1), 47–52 (January 1941).
5. Grubbs, Frank E. (1950). "Sample criteria for testing outlying observations". Annals of Mathematical Statistics **21** (1): 27–58. doi:10.1214/aoms/1177729885.
6. ISO 17034:2016(E) General requirements for the competence of reference material producers.

Revision History

This is a new version of the current SOP.
Revision 1: Clarification on ISO standards being followed. A section was added to address appropriate labelling and packaging of PTs and RMs. Statistical analyses were revised and corrected. KLF and CKM 3/6/2017
Revision 2: Improvement on readability. MKR 5/9/2017

Appendix A Cochran Values

Number of Labs	Cochran Value
2	0.999995503
3	0.99820081
4	0.986411961
5	0.962294008
6	0.930101322
7	0.894205906
8	0.857467523
9	0.821493141
10	0.787100641
11	0.754652554
12	0.724259499
13	0.695896557
14	0.66947017
15	0.644855411
16	0.62191635
17	0.600517091
18	0.580528364
19	0.561828985
20	0.544307847
21	0.527864326
22	0.512406514
23	0.497852062
24	0.484126332
25	0.471162467
26	0.458899909
27	0.447283905
28	0.436265327
29	0.425799576
30	0.415846164
31	0.406368491
32	0.397333262
33	0.38871011
34	0.380471365
35	0.37259179
36	0.365048242

37	0.357819486
38	0.35088611
39	0.344230101
40	0.337835093
41	0.331685772
42	0.325768156
43	0.320069178
44	0.314576818
45	0.309279852
46	0.304167907
47	0.299231383
48	0.294461198
49	0.289849003
50	0.285386907
51	0.28106762
52	0.276884304
53	0.272830531
54	0.268900253
55	0.265087839
60	0.247618352
70	0.21906371
80	0.196679892
90	0.17863704
100	0.163767481
110	0.151290312
120	0.140663114
130	0.131496903
140	0.123505359
150	0.116472967
160	0.110234244
170	0.104659954
180	0.099647789
190	0.095115446
200	0.090996092
210	0.087234948
220	0.083786438
230	0.08061258
240	0.077681279

250	0.074965364
300	0.063898255

Appendix B Grubbs Values

N	TValue	GValue
3	707.3548314	1.154699384
4	38.47052963	1.4989875
5	15.90906487	1.778345973
6	10.58980772	2.005783308
7	8.428168928	2.191954086
8	7.297841726	2.346238637
9	6.615345272	2.47598776
10	6.163418149	2.586682873
11	5.844438958	2.682368944
12	5.608557762	2.766049702
13	5.427842364	2.839984391
14	5.285499007	2.905897765
15	5.170856734	2.965126482
16	5.076826373	3.018721427
17	4.998522692	3.067519948
18	4.932474565	3.112197522
19	4.876150744	3.153305235
20	4.827663735	3.191297358
21	4.785578577	3.226551901
22	4.748785693	3.259386147
23	4.716414178	3.29006853
24	4.687771304	3.318827811
25	4.66229947	3.345860255
26	4.639545012	3.371335296
27	4.619135235	3.395400046
28	4.600761254	3.418182918
29	4.584165007	3.439796558
30	4.569129298	3.460340238
31	4.555470086	3.479901827
32	4.543030446	3.498559407
33	4.531675799	3.516382634
34	4.521290114	3.533433864
35	4.511772847	3.549769108
36	4.503036468	3.565438834

37	4.49500445	3.580488655
38	4.487609608	3.594959907
39	4.480792734	3.608890157
40	4.474501465	3.622313628
41	4.468689331	3.635261572
42	4.463314961	3.647762598
43	4.458341415	3.659842945
44	4.453735612	3.671526736
45	4.449467847	3.682836187
46	4.44551138	3.693791799
47	4.441842079	3.704412524
48	4.438438116	3.714715913
49	4.435279703	3.724718245
50	4.432348866	3.734434644
51	4.42962924	3.743879184
52	4.427105903	3.753064977
53	4.424765219	3.762004261
54	4.422594708	3.770708469
55	4.420582927	3.7791883
60	4.412565056	3.818551768
70	4.403907353	3.885048477
80	4.401332313	3.93957909
90	4.402291407	3.985524619
100	4.40538488	4.025050065
110	4.40979547	4.059616352
120	4.415023485	4.090250994
130	4.420752414	4.117701358
140	4.42677627	4.142526362
150	4.432958235	4.165153877
160	4.439206089	4.185918052
170	4.445457097	4.205084364
180	4.451668422	4.222866923
190	4.457810883	4.239440704
200	4.463864808	4.254950375
210	4.469817217	4.269516805
220	4.475659883	4.283241928
230	4.481387975	4.296212436
240	4.486999096	4.30850263
250	4.492492593	4.320176631
300	4.5182426	4.371028897