Universität Stuttgart
Germany

EAC PT evaluation workshop

Arusha
3-4 February 2009

Report on the EAC Proficiency Testing Evaluation Workshop with Training Course on Quality Assurance in Analytical Chemistry

Arusha, Tanzania, 3 – 4 February 2009

Prepared by Dr.-Ing. Michael Koch

Summary

The workshop covered the evaluation of the 3rd Proficiency Testing rounds on the analysis of edible salt, wheat flour and edible vegetable oil, provided in 2008.

The results showed that in some cases the results from the different laboratories are not comparable, which led to problem that no consensus values could be used as assigned values for the PT.
For other parameters the standard deviation is still to high, so that improvement of the analyses is urgently needed. To facilitate this discussions on the methods used were included in the working group discussions.
The training covered the basics of QA in Analytical Chemistry.

Most of the participants are enthusiastic and very keen to improve their analyses. As the result of the evaluation questionnaire shows, the laboratories benefit very much from a exchange with colleagues form other labs.

Introduction

The workshop reported here followed the 3rd PT rounds of the EAC PT schemes for edible salt, wheat flour and edible vegetable oil (since there was a change from drinking water to edible oil, this was the first round on edible oil).
The PT rounds were organized with support from Physikalisch-Technische Bundesanstalt in Germany.
The workshop covered the report from the experiences of providers as well as participants and the evaluation of the results.
Besides this the opportunity of the workshop was used to provide a training course.

Participants and Organisation

The workshop was attended by 28 participants from the following countries:

- Kenya 13
- Tanzania 9
- Uganda 4
- Rwanda 1
- Malawi 1

In addition the three PT providers and the consultant from Germany were present.
A complete list of participants is given in annex 1.

Tuesday, 3 February 2009

9:00 h Opening – Phenny Kaviiri and Willy Musinguzi
WM put emphasis on the importance of SQMT (Standardization, Quality Assurance and Accreditation, Metrology and Testing) in the region.

9:30 h Introduction of participants

9:40 h Why PT? – background and objectives – Felista Kerubo Nyakoe (KEBS)
The importance of quality and quality management in the laboratory was explained.
Tools and procedures for quality management were described:

- Uniform procedures in the lab
- Control charts
- Reference materials
- Proficiency Testing

Proficiency tests are especially important in the laboratory on the background of the requirements of ISO/IEC 17025.
She explained how PTs are provided by the organizers (preparation of samples, assessment of data etc.).
She described the benefits of participation in PT and gave tips for better PT results.
The complete presentation is included in annex 2.

10:30 h Coffee/tea break

10:50 h Report on Edible Salt PT – Edith Lyimo (TBS)
EL explained the preparation of the samples (mainland salt, initial amount 20 kg, 60 samples were made and sealed immediately).
Parameters to be analysed were:

- NaCl
- Mg
- Ca
- I
- Sulphate
- Moisture

49 samples were dispatched, 21 labs reported results.
The robust mean of all participants' results (consensus mean) was used as assigned value where possible.
Z-scores were used for the assessment of the results. She presented the results of all parameters. The evaluation of the sulphate determination was not possible since there was no consensus between the participants and so no assigned value could be determined.
There was a lively discussion following the presentation (annex 3).

11:30 h Report on Wheat Flour PT - Anthony Irungu (KEBS)
AI also reported on the preparation of the samples. They were mixed manually. Packages for each participant contained approx. 100g. Homogeneity was checked. 31 samples were sent out, 28 participants returned results. Evaluation was made according to ISO 13528.
Parameters to be analysed:

- Moisture

- Total ash
- Crude fat
- Crude protein
- Acidity as lactic acid
- Acidity as sulphuric acid

AI presented the results and the challenges. Acidity as lactic acid and acidity as sulphuric acid could not be evaluated, because there was no consensus between the participants.
The full presentation is given in annex 4.

11:55 h Report on Edible Oil PT – Phenny Kaviiri
PK also described the preparation of the samples (sunflower oil, spiked with Cu and Ni). Manual mixing was done by shaking. Samples were kept at 20°C. Homogeneity and stability were checked. 27 samples were sent out, 22 labs returned results.
Parameters to be analysed:

- Rel. density
- Refractive index
- Acid value
- Iodine value
- Peroxide value
- Copper
- Nickel
- Moisture and volatiles

PK presented the evaluation procedure and the results. The results from the determination of moisture and volatiles as well as copper and nickel could not be evaluated since there was no consensus between the participants. The full presentation is given in annex 5.

12:30 h Assigned values and reference measurements – M. Koch
MK presented the problems with the assigned values using a consensus mean. If there is no consensus between the participants or if the mean of all participants is suspect to be wrong, the mean values of all participants is not a good and reliable estimate of the true value. In these cases reference measurements could be an alternative. If these measurements are traceable to a reliable reference (exact realization of the SI unit or just a reference method), this would enable the participants to check the trueness of their measurements results.
The EAC providers are in contact with AFRIMETS (Ms. Sara Prins as chair of the “metrology in chemistry” group of AFRIMETS). It will be tried to identify suitable reference labs within Africa to provide reference values for the next PT round.
The presentation of MK is given in annex 6.

12:50 h Introduction to WG discussion – M. Koch
MK introduced the questions for the working group discussions in the afternoon (annex 7)

13:15 h Lunch break

14:35 h Working group discussion

In the working groups the guiding questions were discussed and the answers presented to the plenary. The following gives the summary of that answers:

Working Group Questions – Edible Salt

1. *Did you receive the samples in good time and good state?*
   Yes
2. *Was the time given for analysis enough?*
   Yes
3. *Was the sample quantity adequate?*
   Yes
4. *Was the information given sufficient?*
   Some: not sufficient (result sheet not clear)
5. *Did you understand the PT report? –*
   Is there something more to be included?
   Yes, understood
   Recommended to name all labs participating
6. *Was the participation helpful?*
   Yes
7. *Any suggestions for the next round (e.g. parameters)?*
   Expand to nutrients in animal feeds
   Building capacities for pesticide residues
8. *Is a fee of 100 US-$ affordable?*
   Yes, but method of payment is expensive, maybe easier via local coordinators or for several rounds to reduce bank fees
9. *For salt PT:*
   - *Calcium – no real consensus - problems in the method?*
     Maybe better to recommend a method
   - *Sulphate – no real consensus - problems in the method?*
     Maybe better to recommend a method
   - *Iodine – some values are very high, some very low - problems in the method?*
     Different problems in the method. Reagents have to be properly standardized
   - *NaCl – Does this parameter make sense in future PTs?*
     It makes sense and is needed. Recommendation to analyse chloride and call it “Chloride as NaCl”.
   - *Moisture determination at different conditions (temperatures: 105°C, 110°C, 130°C, 250°C)*
     Guideline to be given by the provider

Working Group Questions – Wheat flour

1. *Did you receive the samples in good time and good state?*
   Yes
2. *Was the time given for analysis enough?*
   Enough
3. *Was the sample quantity adequate?*
   Yes
4. *Was the information given sufficient?*
   Yes

5. *Did you understand the PT report? –*
   Is there something more to be included?
   Generally yes, understood
6. *Was the participation helpful?*
   a) helped the organisation in ISO accreditation
   b) enhanced customer satisfaction
   c) analysts have more confidence in their equipment
   d) a plus for donors
   e) helps in continuous improvement
   f) in-house-comparison of analysts
7. *Any suggestions for the next round*
   (e.g. parameters)?
   Expansion of scope: milk? forage? juices?
   Expansion of whet flour parameters: aflatoxin, fibre, acid in soluble ash, microbiology
8. *Is a fee of 100 US-$ affordable?*
   Yes, communication to accompany the payment
9. *For wheat flour PT:*
   - *There are at least three methods to measure acidity and a lot of confusion*
     - Aqueous extraction – reporting usually as lactic acid
     - Ethanolic extraction – reporting usually as sulphuric acid
     - Light petroleum extraction – reporting as free fatty acids
   - *What makes sense? Which parameter should be included in the next round? Who is using which method? Is there a need for harmonization*
     Pearson methods were mostly used.
     Possible factors leading to wrong results:
     - Titrant concentration (not standardized) could be a problem
     - Sample handling: possibly fermentation process
     - Provider to prescribe time
     - Glassware cleaning
     - Temperature could affect the result (water bath 40°C)
     - In future: provider should strongly recommend a single method, provider to distribute the method

Working Group Questions – Edible Oil

1. *Did you receive the samples in good time and good state?*
   Yes
2. *Was the time given for analysis enough?*
   Yes
3. *Was the sample quantity adequate?*
   Yes, but some labs would like to have more sample
4. *Was the information given sufficient?*
   Yes
5. *Did you understand the PT report? –*
   Is there something more to be included?
   Yes, understood
6. *Was the participation helpful?*
   Yes
7. *Any suggestions for the next round (e.g. parameters)?*

PT provider to recommend methods

8. *Is a fee of 100 US-$ affordable?*
   Yes
9. *For edible oil:*
   - *Moisture and volatiles – two different approaches in the standard – which one is commonly used?*
     The oven method is most widely used, but some use hot plate or IR
     Most probably problems occurred with autoxidation
   - *Peroxide value – are there other methods than ISO 3960 used?*
     Titration method, method is highly empirical
   - *Acid value – problems in the method (solvent, hot or cold)?*
     Unit problems, most labs used ISO method
   - *Copper and nickel – problems in the method?*
     Some labs used digestion prior to analysis; some other direct method according to ISO (both AAS)

   A reference lab would be highly appreciated

17:45 h Wrap-up and end

Wednesday, 4 February 2009

Training on "Quality Assurance in Analytical Chemistry"
M. Koch gave a full-day training on the mentioned topic including some exercises. The full presentation is included in annex 8. Because of time constraints the last part of the training (measurement uncertainty estimation) had to be omitted.

Evaluation questionnaire
M. Koch distributed an evaluation questionnaire (annex 9) for the workshop to be filled out by all participants.
The results of this questionnaire were as follows:

The venue of the workshop

Very good:	12
Good:	16
Fair:	2
Poor:	0
Very poor:	0

mean: 1.67 (1 for very good, 2 for good, 3 for fair, 4 for poor, 5 for very poor)

The content of the presentation

Very good:	20
Good:	9
Fair:	
Poor:	
Very poor:	

mean: 1.31 (1 for very good, 2 for good, 3 for fair, 4 for poor, 5 for very poor)

The material distributed:

Very good:	17
Good:	11
Fair:	1
Poor:	
Very poor:	

mean: 1.45 (1 for very good, 2 for good, 3 for fair, 4 for poor, 5 for very poor)

The working group discussions:

Very good:	16
Good:	12
Fair:	1
Poor:	
Very poor:	

mean: 1.48 (1 for very good, 2 for good, 3 for fair, 4 for poor, 5 for very poor)

The evaluation of the PT

Very useful –	1	20
	2	8
	3	
	4	
Not useful –	5	

mean: 1.29

Training:

Very useful –	1	26
	2	2
	3	
	4	
Not useful –	5	

mean: 1.07

Expectations fulfilled:

Yes:	29
No:	0

The most important topics
(the number in brackets gives the number of participants that mentioned this topic):

- Control Charts (22)
- Validation / Calibration (18)
- Quality Management System (13)
- Traceability (11)
- Internal Quality Control (9)
- Proficiency Testing (9)
- Reference materials (8)
- Evaluation of PT round and its importance (7)
- External Quality Control (6)
- Why participate in PT (5)
- Quality assurance in analytical chemistry (5)
- Quality Control (3)
- Group discussions on problematic methods (3)

- Corrective Actions (3)
- True and Conventional values (2)
- Quality and quality goals (2)
- Quality of test results (2)
- Accuracy / trueness / precision (2)
- The interactions (questions) (2)
- Validity of test methods (1)
- Z-score calculations (1)
- Objectives and background of EAC-PTs (1)
- Challenges faced by both provider and participants (1)
- What is Quality (1)
- Control Charts Software (1)
- Selection and calculation for assigned values in PT (1)
- Measurement of Uncertainty (1)
- Glossary (definition of terms) (1)
- All topics (1)

Benefits:

- I had an opportunity to meet and discuss lab testing in this African region with peers
- I have realized major areas where as a laboratory we have to put more emphasis, like the use of control charts, use of CRM
- Networking and understanding more on principles of analysis and quality measurements. Z-score system and review of results. Thank you for sponsorship. Maybe in future PT should be localized regionally – Many thanks!
- I have been able to make connections with people who work in different laboratories. I have learnt how to prepare control charts which is a useful tool for analysis
- The use of control charts
- Developing of control charts, e.g. range charts
- It was quite informative and interactive as we shared on how to improve how we carry out analysis and methods employed. There was comparison on methods and techniques. The contacts made will ensure that my lab can get assistance on various issues during analytical difficulties
- I fully understood the importance of PT. I also pick valuable information of some improvements I can make in our labs
- Technical assistance to my existing problems within my laboratory (i.e. in LOD, LOQ will help to clear my non-conformances)
- How to use the control chart and interpretation of the measured value. To learn how the PT is evaluated and the challenge which occurred during analysis and sample preparation. Understand how to choose the CRM, how to use it in validation and calibration purpose.
- Importance of participating in PT and evaluation. Technique of how to establish control charts
- I got answers for the problems I encountered in the last PT round
- The way to deal with technical quality control system.
- Correctively we can improve the services we offer as technical personnel
- The benefit I got from the workshop is about the use of control charts because for my side I think the control charts (Range- and X-charts) will help to identify

some errors I made in last PT and take appropriate corrective actions for next PT
- More skills in above topics. Good networking
- Some clues of corrective actions on the parameter that failed
- Learning and sharing
- I managed to get test methods on certain parameters
- The training has helped me to improve my work performance and I am sure that next time I will testify the good results I will get from PT
- The relevance of PT and quality management
- Confidence in our lab results.
- Discussion of the PT results and the training component of the workshop. Networking with the consultant, PT provider and the participants
- Networking and experiences from the region. Understood need for good quality management system and how to go about it
- Method improvement guidelines from discussion groups
- Shared experiences and gained hints of probable mistakes, training widened the knowledge in the use of control charts, metrological traceability

Comments:
- The exercise challenging and very valuable
- The interlaboratory interaction sessions should be more encouraged, not only early, but as much as possible, like 3 times a year
- Please the organizers: There is no harm to assist the participants into other issues like reconfirming their tickets so that they can have enough time for the workshop. Please a small allowance
- To be conducted as plan so as to improve our institution. To increase the number of days to three instead of two so that to cover and catch up as the material seems to be many for two days

Report prepared by Dr.-Ing. Michael Koch
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