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Working Material

*National Consultants' Meeting on Inter-comparison  
feedback of Proficiency Tests performed in 2011 and 2012  
for NAA and other Analytical Techniques*

*Report of a Workshop under regional TC project RAF4022*

*Tunis, Tunisia, 4-8 June 2012*

NOTE

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## 1. BACKGROUND

Enhancement of low and medium power research reactor (RR) utilization is often pursued by increasing the neutron activation analysis (NAA) activities. Over the years, the IAEA has stimulated the orientation of NAA groups worldwide on fields of application in which large amounts of samples may exist for analysis. Whereas the markets for NAA laboratories may have been identified, an underestimated problem remains the quality assurance and quality control (QA&QC), which limits tremendously the commercial routine application of this powerful technique.

In 2010, the IAEA facilitated participation of laboratories, involved in IAEA/AFRA project RAF/4/022 (Enhancement of Research Reactor Utilization and Safety), in analytical proficiency testing organized by IAEA in conjunction with WEPAL, the Wageningen Evaluating Programs for Analytical Laboratories ([www.wepal.nl](http://www.wepal.nl)) to assess their analytical performances. WEPAL, a provider of Interlaboratory comparison schemes, is accredited by the Dutch Council for Accreditation, for compliance with the International Standard ISO17043:2010. The Proficiency Testing rounds were related to the determination of (trace) elements in soil and plant materials. Two such rounds (IPE/ISE 2010-3 and -4) have been organized, and the results evaluated by the IAEA supported by Experts in nuclear and non-nuclear analytical techniques. This evaluation was subsequently discussed during an Intercomparison feedback meeting held in Antananarivo, Madagascar from 12 to 16 September, 2011. Potential sources of error, technical and organizational, were identified when possible for the individual laboratories, and action plans for improvement were drafted and accepted. All participants highly valued the outcome of the feedback meeting, and they recommended that the Agency continued with this approach of proficiency testing and evaluation.

Consequently, two new Proficiency Testing rounds have been organized by the IAEA, again in conjunction with WEPAL, to assess the analytical performance of laboratories participating in the IAEA/AFRA project RAF /4/022. The Proficiency Testing rounds with reporting deadlines of 31 December 2011 and 31 March 2012, also related to the determination of (trace) elements in soil and plant materials, were held (WEPAL Codes ISE 2011/4 and IPE 2011/4 and (WEPAL Codes ISE 2012/1 and IPE 2012/1, respectively). The activity within IAEA/AFRA/RAF 4/022 coincided with similar assessments amongst European and Latin American analytical laboratories under IAEA TC projects RER1007 and RLA0037 respectively.

The results of the proficiency testing rounds were again evaluated by Experts to the IAEA and a follow-up Intercomparison feedback Meeting was held in Tunis (Tunisia) from 4 to 8 June 2012. The 18 participants were representatives of research reactor (RR) operating facilities and analytical laboratories in 9 African Member States (MSs). Of these, 6 are with operational RRs: Algeria, Egypt, Ghana, Morocco and South Africa; the other 3 MSs were: DRC (RR not operational), Libya (RR not operational) and Sudan (no RR, only planned). Participants from Nigeria (operational RR), Cameroon and Madagascar (no RR) could not attend the meeting whereas participants from Tanzania and Tunisia (both without RRs) attended the meeting though not having participated in the proficiency testing rounds. The IAEA was supported in the evaluation and discussion of the results by two Experts from The Netherlands (Mr. Peter Bode) and Austria (Ms. Elisabeth Zeiller – who could not attend the meeting), and the meeting was shared by IAEA staff member Mr. Danas Ridikas (IAEA/NAPC). The meeting agenda and a complete list of participants are provided in Annexes I and II.

## 2. OBJECTIVES OF THE WORKSHOP

The objectives of this meeting were:

- a) To analyze critically the performance of the laboratories participating in said proficiency testing rounds.

- b) To assess critically the effectiveness of the correction actions taken after the Inter-comparison Feedback meeting in September 2011.
- c) To identify major problems encountered, discuss potential sources of error and associated solutions for improvement.
- d) To provide training in quality control and quality assurance and other analytical aspects of NAA and other analytical techniques.
- e) To establish action plans for improvement and renewed assessment of the analytical performance via proficiency testing rounds.

### 3. WORK DONE

The participants were welcomed by Mr Mourad Telmini, Director General of National Center of Nuclear Science and Technology (CNSTN), Tunisia. The opening ceremony also included addresses by Mr Nafaa Reguigui, National Coordinator IAEA/AFRA/RAF 4/022 project, Mr Peter Bode, Expert to the IAEA and Mr Danas Ridikas, IAEA representative.

The meeting's agenda (see Annex I) was adopted by the participants. Mr Ridikas gave an overview of IAEA's activities related to enhancement of research reactor utilization and highlighted the overall objective of IAEA/AFRA RAF 4/022 and the Proficiency Testing Rounds organized from 2011 and 2012. He explained that, unfortunately, the samples for the WEPAL round 2011-4 were dispatched rather late due to procedural problems between IAEA and WEPAL and expressed his appreciation that still almost all participants were able to submit results timely before the (WEPAL extended) deadline.

Mr. Bode gave an overview of all the PT results of all participants that led to the conclusion that the degree of trueness in most laboratories during the rounds in 2010 did not correspond with the current state-of-the-practice of the analytical techniques employed. He summarized and presented this based on qualitative performance indicators as depicted in Annex III. Mr. Bode concluded that several laboratories reached the state-of-the-practice of NAA with respect to the degree of trueness typical for these soil and plant matrices such as Laboratory 1029, MASHA-Algeria. Laboratories 1032 GAEC-Ghana, 1034 CERT-Nigeria, 1035 NECSA-South Africa and 1038 PETRO-Sudan did so by significant improvement of their performance compared to their performance in the rounds ISE/IPE 2010-3/4. The other laboratories also improved their degree of trueness compared to the rounds in 2010 but did not reach the quality that normally would be attainable for the components measured. The participant from Laboratory 1037 Libya explained that no analytical activities could be deployed until now due to the political situation in the country (RR is not operational at the moment), but there is a possibility that this may become possible again later in the year 2012.

The individual participants presented – following a template provided by the IAEA prior to the meeting – details of their analytical methodology applied in the Proficiency Testing rounds and their own evaluations of their performance. Each presentation was followed by a discussion, moderated by the Expert, to identify the most significant source(s) of error and for exploring opportunities for improvement. Participants also gave an overview of the status of the implementation of the planned corrective actions drafted in September 2011, thus emphasizing the effectiveness thereof. The feedback on the proficiency testing rounds was completed by conclusions of the participants on the relevance and impact of these rounds organized in 2011 and 2012 (Annex IV), and with recommendations for follow-up. The participants concluded that the Agency's continuous support to the African MSs is still essential for human resource development and capacity building in the analytical laboratories.

Mr. Ridikas commented that several participants asked the Agency for new reference materials and standards but that these were not supplied in all cases. He indicated that the time between the feedback meeting in September 2011 and the PT rounds 2011-4 and 2012-1 was too short to implement all such

support. Meanwhile, countries like Ghana, Morocco and Nigeria on request have received expert missions to advise them on NAA procedures and methodology.

At the end of the meeting, the host, the National Center of Nuclear Science and Technology, organized a technical tour to their facilities, which was appreciated by the meeting participants. The participants were guided through the operations of the new electron beam gamma-sterilization plant, the spectroscopy laboratory for alpha, gamma and X-ray fluorescence analysis and the neutron activation analysis laboratory with the new D-D-neutron generator.

### **3.2 Lectures on sources of error in analytical techniques**

The Expert provided a group exercise on evaluation of PT results and a series of lectures on implementation of quality management activities like internal quality control, use of controls charts and metrological aspects of (nuclear) analytical techniques (such as evaluation of the uncertainty of measurement, and method validation). In addition, an outline was given on steps to be taken towards laboratory accreditation since most of the participants expressed the intention of doing so (see the meeting agenda in Annex 2). The information content of these presentations, with some supporting documentation, has been made available to all participants.

### **3.3 Follow-up activities**

Participants, following the discussions with the Expert, formulated their action plans (Annex V) for the period June–October 2012. It was agreed that once these new remedial and corrective actions have been implemented, participants will re-analyse the PT samples distributed in the WEPAL ISE/IPE 2010-3 round. This will provide them an immediate indication of the effectiveness of their improvements and the growth in performance. These re-analyses will be completed before December 22, 2012 and used for internal evaluation only (e.g. by the IAEA and Experts).

IAEA and participants appreciated the initiative of the Algerian participant for inviting an additional laboratory in Algeria (1110 NOUSSE) to participate in the 2011-4 and 2012-1 rounds and laboratories in Tanzania and Tunisia for their interest in this PT activity in the RAF 4/022 project. They all look forward to their results of new PT analyses.

The IAEA representative, Mr. Ridikas, outlined the IAEA's capabilities to facilitate again, in conjunction with WEPAL, new Proficiency Testing Rounds for African analytical laboratories. This activity may coincide again with a similar assessment amongst European and Latin American analytical laboratories. Taking into consideration the re-analysis of the WEPAL ISE/IPE 2010 samples, the new Proficiency Testing round is foreseen for the period January–March 2013 (WEPAL codes ISE 2013/1 and IPE 2013/1). All these activities will be followed-up by a new feedback meeting around May–June 2013, possibly in Vienna, Austria at IAEA HQs.

## **4. DISCUSSION: SOURCES OF ERRORS**

During the meeting participants and Expert identified several potential sources of error that may have caused unsatisfactory performance in the Proficiency Testing rounds; remedies have been discussed too. These evaluations have been summarized in the Table below.

<b>Potential Source of Error</b>	<b>Remedy Suggested</b>
Improper sample treatment and processing  No correction for moisture content	Use minimum sample mass at the same level as the minimum mass recommended in the certificate of the RM applied for internal quality control  Adhere strictly to the instructions of the PT provider
Insufficient insight in the quality of digestion methods applied	Adhere to Experts' advice in the 2011 Feedback Meeting on sample digestion methods; eventually, contact IAEA expert for help
Inappropriate calibration of facilities	Use calibrators following instructions of the calibrator provider (e.g. (C)RM provider); Training; Assessment of neutron fluence gradients and geometrical factors during counting; Attention to commutability of calibrators (specially for XRF and techniques needing digestion); Attention for blank and background correction; Calibration of balances, pipettes and other utilities; Introduction of performance tests.
Missing suitable calibrators and (certified) reference materials used	Ask Agency's assistance in procurement of new calibrators, (certified) reference materials and standards; Storage and usage following the associated instructions to avoid contamination and deterioration and evaporation losses; Introduction of trend control charts to verify their stability
Inadequate in-house training and qualification, resulting in insufficient awareness on sources of error and sources of uncertainty of measurement	Expert missions and thematic workshops
Inadequate internal quality control throughout the entire analytical procedure, including the assessment of blank and background contributions	Use of internal quality control materials (such as reference materials and blanks) with each batch of samples analysed; Formulation of acceptance criteria and control charts for trend analysis
Insufficient time for cross-checking of the final results and inappropriate reporting; as well as the internal distribution of the PT reports. Wrong units for reporting data, exchange of results and sample codes.	Implement a procedure for planning of analyses, based on reverse planning from the date of reporting; Assuring supervision and cross-checking of results prior to reporting; Adhere to PT provider's instructions for reporting; Contact the IAEA if PT reports are not received within 1 month after the provider's deadline date.

## 5. RECOMMENDATIONS

The participants make the following recommendations, forthcoming from their experiences in these and previous Proficiency Testing Rounds and this feedback meeting:

### To the Laboratories participating in RAF/4/022

1. Adhere to the remedies identified from analytical and organizational improvement of the performance of analytical laboratories.
2. Distribute and study the information made available by the Expert in this meeting.
3. Implement QC/QA practices, including training.
4. Continue participation in new PT through WEPAL ISE/IPE 2013-1.
5. Reminders of the samples of ISE/IPE 2010-3 will be sent by the participant of DRC to the participating laboratory in Tanzania before August 1, 2012.
6. Implement remedial and corrective actions before Oct 15, 2012, if possible.
7. Re-analyse ISE/IPE 2010-3 samples. Report (using the WEPAL-forms) before deadline Dec 22, 24:00; report ONLY to IAEA-TO (Mr. Danas Ridikas) (not to WEPAL!!!!).
8. Provide feedback to IAEA Technical Officer (Mr. Danas Ridikas) on RR utilization in RAF/4/022 when encountering unanticipated limitations and (extreme) hindrances that make execution of the action plan impossible.
9. Communicate with Experts on specific technical problems.

### To the Governments of the African MSs participating in RAF/4/022

1. Consider and facilitate requests for human capacity development in analytical techniques using national resources and/or via support by the IAEA.
2. Provide appropriate support to cover the running and modernization costs of participating analytical laboratories.
3. Expand, together with the participating laboratories, the national awareness on the impact of analytical measurements and the potentials of the national laboratories.

### To the IAEA

1. Facilitate continuation of PT via WEPAL for the new round 2013-1 and coordinate internal to the project PT with already available samples.
2. Inform participants on the dispatch dates of PT samples and associated PT reports.
3. Evaluate results of re-analysis and report on it to the participants before February 1, 2013.
4. Organize new Inter-comparison Feedback meeting May/June 2013 in Vienna, Austria.
5. Strongly consider inviting, if still appropriate, the same participants and Experts in the next feedback meeting as in the meetings held from September 12 to 16, 2011 in Antananarivo, Madagascar and June 4-8, 2012 in Tunis, Tunisia, to strengthen on communications and collaborations amongst them established during these meetings, and to ensure a balanced evaluation of the effectiveness of action plans.
6. Facilitate provision of calibrators and reference materials, and expert missions on request.
7. Consider support in organizing regional analytical workshop periodically.
8. Consider and coordinate a scientific publication in an internationally reviewed scientific journal, emphasizing the methodological approach and outcome.
9. Find an approach for support in transnational/regional access to RR facilities.

### To the Experts

1. Evaluate the performance of the laboratories in the next PT rounds (old and new) within 2 weeks after their availability, and report on them to the IAEA TO.
2. Provide advice to participants on analytical deficiencies encountered.
3. Suggest, in conjunction with the participants, a series of appropriate lectures for the next feedback meeting.
4. Assess if a scientific publication may be drafted on basis of the outcome of this approach for PT testing and feedback meetings.

## 6. CONCLUSIONS

Participants agreed that a Proficiency Testing with associated feedback meeting has proven to be an effective mechanism for assessment of performance as well as identification of sources of error and to obtain pragmatic solutions for improvement. They concluded that this approach has been very helpful, and therefore recommend the IAEA to continue this mechanism in future Proficiency Testing rounds amongst laboratories that are building and strengthening their analytical performance.

**Several laboratories reached the state of the practice of NAA with respect to the degree of trueness typical for these soil and plant matrices such as Laboratory 1029, MASHA-Algeria. Laboratories like 1032 GAEC-Ghana, 1034 CERT-Nigeria, 1035 NECSA-South Africa and 1038 PETRO-Sudan did so by significant improvement of their performance compared to their performance in the rounds ISE/IPE 2010-3/4. Some improvement is also visible for soil and plant parameters other than trace elements in the results of laboratory 1036 LASPEE-Cameroon. The remaining participating laboratories also improved their degree of trueness compared to the rounds in 2010 but did not reach the quality that normally would be attainable for the components measured. Potential sources of error have been identified during the discussions, thus providing a good basis for substantial improvement in the future of the performance of the other laboratories.**

IAEA and participants appreciated the initiative of the Algerian participant for inviting additional laboratories in Algeria (e.g. 1110 NOUSSE) to participate in the 2011-4 and 2012-1 rounds and laboratories in Tanzania and Tunisia for their interest in this PT activity in the RAF 4/022 project.

The participants were satisfied with the additional series of lectures on sources of error in analytical techniques as it provided complementary information needed for further analytical improvement.

The meeting resulted in individual action plans to be implemented – with appropriate assistance from the IAEA – in the laboratories before the planned re-analysis of the samples from the 2010-3 PT round, to be reported ultimately to the IAEA by December 22, 2012.

Participants concluded the meeting with intensive discussion and sharing of experience on the conduct of analysis with similar techniques, marking the start of more network activities amongst their laboratories. Such a network would benefit from a periodic regional workshop on analysis using nuclear and nuclear-related analytical techniques. In addition, this network could act as a platform for facilitating trans-regional access to RR facilities in particular for countries without or considering the first RR.

**At large, participants agreed that the objectives of the IAEA in facilitating these PT rounds under the project RAF/4/022 were achieved.** The participants, therefore, strongly recommend that the IAEA continues to facilitate new PT Rounds with feedback meetings for independent assessment of performance and the effectiveness of remedial and corrective actions of analytical laboratories.

## ANNEX I. MEETING AGENDA

### National Consultants' Meeting on Inter-Comparison Feedback of NAA and other Analytical Techniques Proficiency Tests Tunis, Tunisia, 2012-06-04 - 2012-06-08

#### **Monday, June 4**

##### **Morning**

9:00-9:10 Opening of the Meeting by **Host Country and IAEA representatives**

9:10-9:40 Self-Introduction of **participants**: scientific background & function in the institute, max. 2 minutes each

9:40-10:00 Introductory Presentation by **Mr Danas Ridikas** (IAEA)

Coffee break

10:30-11:30 Lecture: Presentation of PT results (by elements and/or methods) by **Mr Peter Bode** (The Netherlands)

11:30-12:30 **Presentations\*** by **each country** on the methods used for PT sample measurement: Information on sample preparation, measurement details and Quality Control applied. (2 countries; ca. 30 minutes each) as well as their own follow-up activities (root cause analysis of deficiencies and corrective actions, if applicable).

##### **Afternoon**

14:00 – 15:30 **Presentations\*** (Continued) by **each country** on the methods used for PT sample measurement: Information on sample preparation, measurement details and Quality Control applied. (3 countries; ca. 30 minutes each) as well as their own follow-up activities (root cause analysis of deficiencies and corrective actions, if applicable).

15:30-16:00 Coffee break

16:00 – 17:30 **Presentations\*** (Continued) by **each country** on the methods used for PT sample measurement: Information on sample preparation, measurement details and Quality Control applied. (3 countries; ca. 30 minutes each) as well as their own follow-up activities (root cause analysis of deficiencies and corrective actions, if applicable).

***\*Strictly according to the provided presentation template***

**Tuesday, June 5**

**Morning**

8:30- 09:30 **Presentations\*** (Continued) by **each country** on the methods used for PT sample measurement: Information on sample preparation, measurement details and Quality Control applied. (2 countries, ca. 30 minutes each) as well as their own follow-up activities (root cause analysis of deficiencies and corrective actions, if applicable).

*\*Strictly according to the provided presentation template*

09:30-10:00 Coffee break

10:00 – 10:30 Lecture: Lessons to be learned from PT reports by **Mr Peter Bode** (The Netherlands)

10:30-12:30 Discussion on the applied methods, review of experimental conditions and potential sources of errors and pathways to improvements (if possible participants shall allow an identification of their anonymous results to allow judgment of the quality of method) (**All participants and experts**)

**Afternoon**

14:00 -15:00 Drafting conclusions on these PT for meeting report.

15:00-15:30 Coffee break

15:30-16:30 Exercise: Responding to PT results by **Mr. Peter Bode** (The Netherlands)

16:30 – 17:00 Lecture: Simple statistical tools for objective quality assessment and outlier tests by Mr. **Peter Bode** (The Netherlands)

**Hospitality Event**

**Wednesday, June 6**

**Morning**

8:30-09.30 Lecture: Lecture: Internal quality control by **Mr Peter Bode** (The Netherlands)

09:30-10:30 Lecture: Instrument performance assessment by **Mr. Peter Bode** (The Netherlands)

10:30-11:00 Coffee break

11:00- 12:30 Lecture: Control charts by **Mr Peter Bode** (The Netherlands)

**Afternoon**

14:00-15:00 Lecture: Uncertainties: bottom-up and top-down approach and examples by **Mr Peter Bode** (The Netherlands)

15:00-15:30 Coffee break

15:30-16:45 Lecture: Uncertainties (cntd), incl. Kragten spreadsheet, and with attention to NAA by **Mr Peter Bode** (The Netherlands)

16:45-17:30 Sources of error in AAS and ICP (on basis of presentations prepared by Ms. Lisa Zeiller, Austria) by **Mr. Peter Bode** (The Netherland)

**Thursday, June 7**

**Morning**

08:30-9:30 Lecture: Method validation: concepts by **Mr Peter Bode** (The Netherlands)

09:30-10:30 Lecture: Method validation: the practice by **Mr Peter Bode** (The Netherlands)

10:30-11:00 Coffee break

11:00-12:30 Lecture: Metrology: how to reach best measurement capabilities by **Mr. Peter Bode** (The Netherlands)

**Afternoon**

14:00-15:00 Drafting conclusions for meeting report.

15:00-15:30 Coffee break

15:30-17:00 Drafting action plans for the meeting report

17:00-17:30 First discussion on recommendations for the meeting report

**Friday, June 8**

**Morning**

08:30 – 10:30 Presentations by **all countries** (max. 10 min/country) on:

- Most likely sources of error in their technique/laboratory; action plans for improvement and independent means for verification thereof.
- Action plans (if applicable) to inform customers on previously reported data that may be doubtful.

10:30-11:00 Coffee break

11:00 – 12:30 Finalization of meeting report and recommendations

**Afternoon**

**14:00- 17:30 Technical tour to CNSTN**

Closing of the Meeting

## ANNEX II. LIST OF PARTICIPANTS

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20	<b>Sudan</b>	<p>Ms Sara Elsheikh  Central Petroleum Laboratories  Elamarat Street 61  KHARTOUM  SUDAN (could not participate)  Tel.: 09019767761  EEmail: <a href="mailto:sarora22s@hotmail.com">sarora22s@hotmail.com</a></p>

21	<b>Tunisia</b>	<p>Mr Nafaa Reguigui  Centre National des Sciences et Technologies Nucléaires (CNSTN)  Nuclear Safety,  Pôle Technologique  B.P. 72  2020 SIDI THABET, Ariana  TUNISIA  Tel.: 00216 71 537509  Fax: 00216 71 537555  EMail: <a href="mailto:n.reguigui@cnstn.rnrt.tn">n.reguigui@cnstn.rnrt.tn</a>  Internet: <a href="http://www.cnstn.rnrt.tn">http://www.cnstn.rnrt.tn</a></p>
22	<b>United Republic of Tanzania</b>	<p>Mr Remigius Ambrose Kawala  Tanzania Atomic Energy Commission (TAEC)  Nuclear Technology, Environmental &amp; Radiowaste Management, 12  Njiro Area Block "J"  P.O. Box 743  ARUSHA  UNITED REPUBLIC OF TANZANIA  Tel.: 00255 272 508 554  Fax: 00255 272 509 709  EMail: <a href="mailto:remigiusrg@yahoo.com">remigiusrg@yahoo.com</a>  Internet: <a href="http://www.taec.or.tz">http://www.taec.or.tz</a></p>
23	<b>Democratic Rep. of the Congo</b>	<p>Mr Kabongo Paul Kavul  Centre d'Expertise d'Evaluation et de Certification des substances  Minerales  Precieuses et Semi-Precieuses  Avenue Mulongo No. 87  LUBUMBASHI, Katanga  DEMOCRATIC REP. OF THE CONGO  Tel.: 00243 993537203  EMail: <a href="mailto:paulkavul@yahoo.fr">paulkavul@yahoo.fr</a></p>
24	<b>Democratic Rep. of the Congo</b>	<p>Mr Kuanda Thomas Solo  Commissariat général à l'énergie atomique (CGEA)  B.P. 868  KINSHASA XI  DEMOCRATIC REP. OF THE CONGO  Tel.: 00243 818132547  EMail: <a href="mailto:solka_thomas@yahoo.fr">solka_thomas@yahoo.fr</a></p>

### ANNEX III. SUMMARY\* OF PROFICIENCY TESTS RESULTS

The following criteria have been used to classify the performance of participating laboratories.

**E = Excellent.** No or only few outlying results, typically most results with  $|z| < 3$

**VG = Very Good.** Majority of results with  $|z| < 3$  but more outlying results, thus not qualifying for category 'E'

**G = Good.** Majority of results with  $|z| < 3$  but about one-third outlying results, thus not qualifying for category 'VG'

**M = Moderate.** Just acceptable; about half the results with  $|z| < 3$ , but the rest is outlying

**P = Poor.** Z-scores over a wide range, only few results  $|z| < 3$

**VP = Very poor.**

Participant code & affiliation	Technique applied	ISE 2010-3	IPE 2010-3	ISE 2010-4	IPE 2010-4	ISE 2011-4	IPE 2011-4	ISE 2012-1	IPE 2012-1
1029 MASHA, Algeria	NAA	VG	VG	E	E	E	E	E	E
1030 CGEA, DRC	ICP-MS	VP	M	M	P	P	P	M	M
1031 ETRR, Egypt	NAA					G	E	G	E
1032 GAEC, Ghana	NAA	M/P	P/VP	P	G	E	E	G	G
1033 CNESTEN, Morocco	NAA	E	VG					M	P
1034 CERT, Nigeria	NAA	VG	G/M	G/M	P	E	E	E	E
1035 NECSA, South Africa	NAA	E	VG	G	P	VG	G	E	G
1036 LASPEE, Cameroon	Various, incl ICP	E{P}*	VG{P}*	VG{VP}*	E{P}*			E{P}*	M(P)*
1037 INSTN, Madagascar	XRF	VP		E		P	G	G	M
1038 PETRO, Sudan	ICP-AES	VP				VG			
1039 LARM, Libya									
1110 NOUSSE, Algeria	NAA					G	E	E	VG
8029 TOUMERT, Algeria	XRF					P	P		

\*Individual and more detailed results of the PT tests were made available to all participants or can be additionally provided from the Secretariat on request.

## **ANNEX IV. CONCLUSIONS OF PARTICIPANTS ON THE RELEVANCE AND IMPACT OF PT ROUNDS IPE/ISE 2011-4 AND 2012-1.**

### **Original objectives:**

1. Independent/external assessment by the IAEA of the quality of the results of analytical techniques for (trace) element determination in laboratories of participants in RAF 4/022.
2. Identification of unanticipated sources of errors.
3. Providing an opportunity for exchange of experiences and advice for improvement via a feedback mechanism.
4. Providing an opportunity to explore limitations in determining non-routinely searched elements and matrices at the current state of the practice in the respective laboratories.

### **Conclusions:**

1. Participating laboratories recognized the importance of the PT rounds for assessing the quality of their analytical activities.
2. Remedial and corrective actions, taken after the feedback meeting in September 2011 in Antananarivo, Madagascar, have resulted in clear improvements in degree of trueness compared to the level in the PT rounds IPE/ISE 2010-3 and -4.
3. Five participating laboratories have now an acceptable performance with the majority of the scores  $z < 3$  in the latest PT rounds.
4. All participating laboratories identified some unexpected analytical deficiencies via their participation in these PT rounds.
5. Participants shared and discussed with Experts their analytical procedures. Potential sources of error were put forward for further evaluation and possible corrective action in the individual laboratories.
6. Participants got a clear understanding of the procedures and methodology of PT rounds, and a mechanism for their own evaluation of the outcome thereof.
7. Participants observed the need for renewal of their calibrators and expansion of their (certified) reference materials.
8. In summary, participants agreed that the objectives of the IAEA in facilitating these PT rounds under the project RAF/4/022 were achieved.

## ANNEX V. INDIVIDUAL WORKPLANS FOR 2012

## i. Algeria

Activity	How	Person in charge	Deadline	QA/QC Measure	Result achieved

1. Make aware technicians and scientists about the results obtained and how to avoid the wrong results; YES
2. Find why some elements like Rb could not be well analysed and make corrections; problems with Maestro?
3. Establish actions plan to improve our practices and method; change the method of assembling of samples
4. Make some experiences to determine irradiation site parameters (thermal, epithermal and fast neutron flux, flux gradient in the capsule) and efficiency calibration of detection installation. NO
5. Change irradiation, decrease collection time to improve measurement and reduce turn around analysis; No
6. In absence of RMs, engage preparation of chemical mono and multi-element and validate them; No (lack of chemical standard);
7. Validate the method by using some wepal samples; Yes
8. Re-analyse plant and soil wepal samples; No
9. Achieve home software to determine element concentration and validate it; in progress
10. Our irradiation pneumatic system is now in operation, determine short half live element in wepal sample and compare with those edited by wepal and validate a method ; problem of transfer of rabbit still exist ; so we can't analyse short half live elements
11. Bring more Algerian laboratories to participate to inter-comparison Runs (CRNB, CRNA) Yes

## ii. Cameroon

Activity	How	Person in charge	Deadline	QA/QC Measure	Result achieved
Distribute and study the information made available by the Expert in the meeting held a Tunis	Organise a meeting	Head of the lab	August		
Analytical procedures	Analyse samples with standard methods	Analysts	Start in July		
Analytical procedures	Used of reference material	Analysts	Start in July		
Calibration of systems	Balances, pipettes, oven	technicians	June and will be done once a year		
Acquire pure elements standards solutions		Head of the lab	August		
Re-analyse ISE/IPE 2010-3/4, 2011 and 2012 samples.		technicians	July, august, September		
Find why some trace elements did not well analyzing and make correction		technicians	July		
Implement remedial and corrective actions		Scientist and technicians	Before October		

## iii. DRC

Activity	How	Person in charge	Deadline	QA/QC Measure	Result achieved
Evaluate PT results and present Tunis lectures	Hold a meeting with the laboratory staff	Kavul, Solo	20 <sup>th</sup> July	Meeting effectively held	
Integrate the systematic use of SRMs and CRMs in the laboratory	Use of previous ISE_IPE materials	Kavul	30 <sup>th</sup> June	IQC measures effectively installed	
Obtain best suitable CRMs, SRMs	<ul style="list-style-type: none"> <li>- Submit a purchase to the IAEA</li> <li>- Submit a purchase request to the CEEC Top Management</li> </ul>	Solo, Kavul	request by 5 <sup>th</sup> July	Purchase order placed  New CRMs, SRMs received	
Use Calibrant ranges in two configurations	Perform 2 dilutions range per sample of ISE_IPE_2012.1 solutions	Umba, Yav	25 June – 7 July	Z score obtained compared to the PT exercise	
Use of suitable reagents, by new standard solutions and new gas cylinders	Check expiry date and order new whenever necessary	Laboratory head	30 <sup>th</sup> July	Valid calibrants, gas available	
Follow LOD per element	Use of blanks for LOD determination	All the laboratory staff	15 <sup>th</sup> August	Adapt calibrant range at least 5 fold the LOD	
Apply complete dissolution for sol samples	Apply protocol of Millestone cookbook	Kahuata, Yav, Umba	27 <sup>th</sup> July	% Dissolution recovery calculated	
Implement moisture content measurement for more than 3 h	Apply moisture content protocol, according to Wepal recommendations (more than 4 hours at 105°C)	Solo, Kahuata, Yav, Umba	30 <sup>th</sup> July	Results expressed versus dry mass	
Introduce triplicate repetition	Pass from 2 to 3 measuring samples per material	Kahuata, Yav, Umba		Precision and accuracy achieved	
Apply reproductibility technique	Apply one analyst principle in the measuring sample preparation	Kahuata, Yav, Umba		Precision and accuracy achieved	
Re-analysis of IPE/ISE 2010	Perform 2 measuring exercises at 2 weeks delay	All the laboratory staff	1 <sup>th</sup> – 30 <sup>th</sup> October	Z score gained	

## iv. Egypt

Activity	How	Person in charge	Deadline	QA/QC Measure	Result achieved
NAA group meeting to discuss the lessons learned from the participation in the 2011-4 and 2012 rounds PT and the shortages and defects in the analysis.	Holding a meeting	NAA group leader.	During July 2012.	The meeting was held.	
Assuring that the QA/AC procedures are applying during the experiment different stages.	Putting an experiment checklist.	QA/AC Officer.	End of August 2012.	The checklist was written.	
Providing new Standard materials.	Requesting new Standard materials	NAA group leader.	End of December 2012.	The standard materials were provided.	
Avoiding samples exchanges.	Putting a standard procedure.	QA/AC Officer.	End of August 2012.	The procedure is applied.	
Re-analyse the samples distributed in WEPAL ISE/IPE 2010-3.	Analyse the samples.	NAA group leader.	22 December 2012.	The samples were analysed.	
Participating in PT foreseen for the period January-March 2013 (WEPAL codes ISE 2013/1 and IPE 2013/1).	Analyse the samples.	NAA group leader.	31 March 2013.	The samples were analysed.	

## v. Ghana

Activity	How	Person in charge	Deadline	QA/QC Measure	Result achieved
Lessons from PT Workshop held in Tunis, Tunisia	Hold a meeting with the Manager of the Nuclear Reactors Centre (NRRC), colleagues in the Utilization (NAA) Group	N.S. Opata/ S.B. Dampare	Tuesday, 19 June 2012	Meeting successfully held	Done. Meeting with the Manager of the NRRC first, followed by the NAA Group
Maintenance of rabbit systems and pressure controlled gauges	Tracking possible leakage of pneumatic transfer systems	Maintenance Team and NAA Group ( Technologists /Technicians/ Analysts)	Wednesday, 27 June 2012	Problem identified via effective collaboration between Maintenance and NAA Teams. Inspection by Head of Maintenance. Test runs successfully performed	Task successfully executed
Calibration of systems and high power voltage systems	Balances, pipettes, detectors, HPV systems, etc.	I.K. Baidoo, A.G. Ampong, N.S. Opata	Thursday, 12 July 2012	Control charts, trend analysis, etc.	Task partly executed; control charts and trend analysis in progress
Test running of standards /reference materials and blanks	Sample preparation, irradiation and evaluation of selected reference materials, standards and blanks	NAA Group, led by N.S. Opata	Friday, 20 July 2012	High degree of accuracy using standards and blanks	In progress
Re-visiting the issue of acceptance criteria	Use of SRMs/CRMs/ standards, and evaluation of data using various acceptance criteria	NAA Group, led by S.B. Dampare	Friday, 27 July 2012	Acceptance criteria developed, and compliance demonstrated by analysts	Not yet
Re-analysing previous PT samples ISE/IPE 2010-3	Sample preparation and irradiation, and data reduction and evaluation by WinSPAN 2010 (Ver.2.10)	NAA Group, led by N.S. Opata and I.K. Baidoo	Monday, 10 December 2012	Improved results over the previously reported results to WEPAL , and good agreement between “new” results and WEPAL acceptable results	Not yet

**vi. Libya, not available**

Requested to provide...

Activity	How	Person in charge	Deadline	QA/QC Measure	Result achieved

## vii. Madagascar

Activity	How	Person in charge	Deadline	QA/QC Measure	Result achieved
PLAN	Organize a meeting with the team involved in analysis (technicians and analysts)	Chair : Head of analytical Department	21/09/11	Lesson learned from WEPAL PT 2010-3 : the use of XRF conventional is not good for trace elements, the team has decided to apply the TXRF method for the measurement.  Question : How to DIGEST correctly ISE and IPE samples? Answer : Lessons from PT Workshop held in Antananarivo	Planning agreed
DO	Calibration of analytical equipment (TXRF and AAS)	- Analysts and technicians	26/09/11 – 07/10/11	Use of pure standard solutions.	Linear calibration curve obtained.
CHECK	Quality Control of analytical methods	- Analysts	12/10/11 – 26/10/11	- Use of Certified Reference Materials (CRM) - Use of the calibration curve mentioned in activity “DO”	Due to the lack of CRM, the use of previous testing samples are necessary. Ex. : PTXRFIAEA02 (lichen) for plant. For soil, SOIL-7 is used.  In general, poor results are got for some analytes : Al, Cd
ACT	Re-analyse the WEPAL PT 2010-3 testing samples (ISE and IPE)	- Analysts	31/10/11 - 25/11/11	Use of the calibration curve mentioned in activity “DO”	Results are more or less improved
ACT	Re-analyse the WEPAL PT 2010-3 testing samples (ISE and IPE)	- Analysts	20/11/12 - 20/12/12	Use of the calibration curve	

## viii. Morocco

Activity	How	Person in charge	Deadline	QA/QC Measure	Result achieved
Asking IAEA for assist us to acquire pure elements standards solutions, CRM and SRM	Via IAEA Projets	K. Embarch/H. Bounouira	In progress		
Validation of the method for the analysis of rare earth elements in phosphates: Interferences problems with the fission products generated by uranium	irradiate uranium standard and a rare earth standard and then determine what the apparent concentration for each microgram/gram of uranium.	K. Embarch/H. Bounouira	14/06/2012		Irradiations have already been done. Calcul of the contribution of the fission products is in progress.
Installing the Compton suppression system	Via IAEA TC- Projets MOR1007	K. Embarch/H. Bounouira	In progress		The equipment is already ordered
Installing the pneumatic transfer system for irradiation with epithermal neutrons	With help of IAEA	NAA group And Reactor group	In progress		In progress
Reanalysing the previous PT samples 2010., 2011 and 2012	Analysis of Wepal (soil and plant) samples	NAA group	Operation has been started 01/07/2012		In progress
Training of the NAA laboratory member staff	Fellowship for Mr H. Bounouira in DELF in Netherlands and fellowship for Mr I. AARAB at TEXAS NAA laboratory in USA	The head of Elemental analysis Unit (Mr. M. Bounakhla)	The fellowship of Bounouira will start on September 17 <sup>th</sup> 2012 and for Mr. Aarab the date is not defined		The contact is already established with the supervisors in Netherlands and in USA

## ix. Nigeria

Activity	How	Person in charge	Deadline	QA/QC Measure	Result achieved
Lessons from the PT Meeting held at Antananarivo, Madagascar	Report the outcomes of the meeting to C.E.R.T. Management and discuss with staff of Reactor Lab.	Y.A. Ahmed and I.B. Mansir	20 September 2011	Management's continued support for Quality assured	YES. The outcomes of the PT discussed successfully and advice on improvements given
Lessons from the PT Meeting held at Antananarivo, Madagascar	Hold meeting with the Reactor Manager and Reactor Supervisor	Y.A. Ahmed and I.B. Mansir	22 September 2011	Reactor Management adopted Madagascar improvement measures for QA/QC	YES. Support for implementation of Madagascar corrective measures obtained
Lessons from the PT Meeting held at Antananarivo, Madagascar	Sensitization on Correctives measures to be taken and circulation of action plan	Y.A. Ahmed and I.B. Mansir	26 September 2011	Staff became aware of sources of errors in routine NAA	DONE. Staff agreed to implement corrective measures
Lessons from Dr. Landsberger (IAEA Expert) visit to the Lab	Meeting with all Reactor staff	Reactor Manger	5 October 2011	Adoption of suggestions made by the IAEA Expert	DONE. Suggestions incooperated into routine activities
Systems Calibration	Calibration of weighing balance and HPGe detector	All Scientist and Technologist involved in Reactor Utilization	10 October 2011	Use of Standard Weights and Control Charts	DONE. Good calibrations were obtained
Re-analysing remaining PTs.(IPE/ISE 2010-3/4) samples and plant ans soil CRM	Samples preparation, irradiation, counting and analysis	Staff in Utilisation Group	11-25 October 2011	Comparing the results obtained with the WEPAL mean values	DONE. Very good results were obtained. with z-score of less than.1.5
Analysis of NIST Plant and Soil Standard Reference Materials Only	Samples preparation, irradiation, counting and analysis	Staff in Utilisation Group	11-25 October 2011	Comparing the results obtained with NIST Values	DONE. Good agreements with NIST values

## x. South Africa

Activity 2011-2012	How	Person in charge	Deadline	QA/QC Measure	Result achieved
Determine moisture content	As prescribed by IAEA	N Seaga	March 2012		Not done
Buy new Ca-salt		N Seaga	-		Ca-salt was new
Testing the cleaning with 10% HNO <sub>3</sub>		N Seaga	October 2012		Not done
Post Mortem	Went through all the data	N Seaga	May 2012		Done
Corrective actions after Post Mortem	Still have to write up and prioritise	N Seaga; MJ Raven	October 2012		No
Activity 2012-2013	How	Person in charge	Deadline	QA/QC Measure	Result achieved
Continue on actionplan of 2011-2012	Post mortem actions to implement	N Seaga; MJ Raven	October 2012		
Order new CRM's	Contact IAEA for standards	MJ Raven	Dec 2012		
Resolve Cd problem	Do irradiations for different samples	N Seaga	October 2012		
Put database in place for all elements	Create database and put old data in	MJ Raven	Feb 2013		
U and Th database and control charts therefore	Create database and keep update	N Seaga	October 2012		
Look for multi-element standards	Go on Internet to suppliers and find out if they prepare special standards for our use	MJ Raven	Feb 2013		

## xi. Sudan

Activity	How	Person in charge	Deadline	QA/QC Measure	Result achieved
Distribution of all documents to all participants involved in this project	Email , CDs	Helmay	09,Jul,2012		
Two groups working in this project (SAEC and CPL) will hold a meeting to discuss the findings and recommendations of the last consultant meeting	Invitation will be send by email	Ammar and Helmay	27,Jun,2012		
Quality control	Ordering of new CRMs and SRMs	Coordinator	Jun,2012		
Include wet to dry ratio in calculation	Moisture content determination	Analysts	Should be done within analysis time		
Re-analysis remainders of PTs IPE/ISE 2010-3	ICP-OES	Analysts, etc	01,OCT2012		

**xii. Tunisia, not available**

Requested to provide...

<b>Activity</b>	<b>How</b>	<b>Person in charge</b>	<b>Deadline</b>	<b>QA/QC Measure</b>	<b>Result achieved</b>

## xiii. Tanzania

**MAIN ACTION PLAN**

1. To participate in the next phase 2013/1 WEPAL PT
2. To participate in reanalysis of 2010 WEPAL PT; report to IAEA
3. To revisit QC procedures/documentation and machine status

**BREAKDOWN**

ACTIVITY/ ACTION	HOW	PERSON IN CHARGE	DEADLINE	QA/QC MEASURE	RESULT ACHIEVED
Share knowledge gained and requirements to WEPAL PT	Meeting with lab staff and PC	Mr. Kawala and PC	June 2012	Meeting done, Reallocation of activities done	WEPAL PT participation
Request Biological standard materials	Request tomato leaves Request Chinese And others	Mr. Kawala PC TO	June 2012	IAEA to deliver the SRM NIST require physical address for sending standard	
Sample receipt WEPAL 2010/3 from DR Congo	By DHL, Individual, any other agreed means	Mr. Kawala and Mr Solo from DRC	June to July 2012	Successful be received	Received
Revisit of Lab performance	<ul style="list-style-type: none"> <li>• QC/QA</li> <li>• Calibration</li> <li>• Remeasurement of Std sample</li> </ul>	All technical scientist	June to August 2012	Control charts be established	
Learning the WEPAL requirements on PT	<ul style="list-style-type: none"> <li>• Visit WEPAL website</li> <li>• Consult/ comm. WEPAL group</li> <li>• Consult TO RAF4022</li> </ul>	All technical scientist  Mr. Kawala	June to August 2012	Meet requirement	
Sample preparation	<ul style="list-style-type: none"> <li>• Based on WEPAL requirement</li> </ul>	Mr. Kawala, Ms. Chuma	August 2012		
Sample measurement and analysis	<ul style="list-style-type: none"> <li>• Using XRF</li> </ul>	Mr. Kawala, Ms. Chuma	August 2012		
Reporting	<ul style="list-style-type: none"> <li>• Based on WEPAL system to IAEA</li> </ul>	Mr. Kawala, Ms. Chuma	Nov 2012		
Receipt of WEPAL sample 2013/1	<ul style="list-style-type: none"> <li>• Notifying IAEA</li> <li>• WEPAL notification</li> </ul>	Mr. Kawala PC TO	Feb 2013 Rpt on 31/3/13		