

# External quality assessment of Algerian neutron activation analysis laboratory

**Lylia Hamidatou, Hocine Slamene, Boussaad Zouranen, Samir Beagaa,  
Mohammed Messaoudi, Djebli Kamel**

**Abstract**— Participation in inter-comparison programs is an important process to enhance the accuracy and precision of the analytical techniques used in the laboratories. The NAA laboratory of nuclear research centre of Birine participates during the first quarter of 2013 by analyzing IPE International Plant-Analytical Exchange and ISE International soil-Analytical Exchange ISE distributed by WEPAL accredited organism. This work was executed in the frame work of RAF/4/022 project, Enhancement of Research Reactor Utilization and Safety by taking part in analytical proficiency testing IAEA in conjunction with WEPAL, the Wageningen Evaluating Programs for Analytical Laboratories. All results were evaluated in this paper.

**Index Terms**— Laboratory, evaluation, NAA method, performance, accreditation, proficiency testing.

## I. INTRODUCTION

The Algerian research reactor (Es-Salam) is a 15 MW heavy water reactor type, operating since 1992. Instrumental neutron activation analysis (INAA) was introduced in 1993 at the laboratory of the neutron activation analysis department, in Nuclear Research Centre of Birine, Algeria. In our research reactor, the  $k_0$ -method was successfully developed using the HØGDAHL convention and WESTCOTT formalism [1, 3]. In 2012, 2013 Hamidatou L et al reported in this paper “ $k_0$ -NAA quality assessment in an Algerian laboratory by analysis of SMELLS and four IAEA reference materials using Es-Salam Research reactor” and “NAA Algerian laboratory evaluation processed by WEPAL and IAEA during 2011-2012” the internal quality control of the  $k_0$ -NAA technique [4, 5]. The concept of QC/QA, internal and external validation is considered as an advanced stage in the life cycle of an analytical method. This technique was used for practical applications in several areas of life such as: nutrition, health related to environmental field, mining and industrial fields [6-7].

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**Lylia Hamidatou**, Nuclear Research Centre of Birine, Algeria. P.O. Box, 180 Ain Oussera, Djelfa, Algeria (e-mail: lylia.h@hotmail.fr).

**Hocine Slamene**, Nuclear Research Centre of Birine, Algeria. P.O. Box, 180 Ain Oussera, Djelfa, Algeria

**Boussaad Zouranen**, Nuclear Research Centre of Birine/ COMENA, Algeria.

**Samir Beagaa**, Nuclear Research Centre of Birine, Algeria. P.O. Box, 180 Ain Oussera, Djelfa, Algeria

**Mohammed Messaoudi**, Nuclear Research Centre of Birine, Algeria. P.O. Box, 180 Ain Oussera, Djelfa, Algeria

Recently, there is a growing interest in the use of the NAA method for trace element determination in the industrial sector. The richness of the service analytics in our NAA laboratory has been in the diversity of solid samples. The dominant method used has been neutron activation analysis at Es-Salam research reactor during 20 years of its activity. The viewpoint is that the neutron activation analysis remains the best tool to determine the contents of trace elements in all types of samples [8, 9].

At Es-salam research reactor, the INAA and  $k_0$ -NAA techniques are fully applied by researchers. In addition, the development of other analytical techniques based on the neutron activation is also achievement such as the cyclic delayed neutron counting technique and the application of RNAA to the proportioning of iodine in food salt [10, 11]. The NAA laboratory work to make an effort to connect the unique features of NAA activities in a strategic way for the national goals. A reactor centre, whether it be in a university or other institution, is a substantial investment in the human and financial resources of a developing nation.

The current status of the NAA method, the analytical procedures and the nuclear activities of our laboratory are presented in the chapter “Concepts, Instrumentation and Techniques of Neutron Activation Analysis” newly published in the book: Imaging and Radioanalytical Techniques in Interdisciplinary Research - Fundamentals and Cutting-edge Applications [9].

A regular independent assessment of the technical performance of a laboratory is necessary to assure the validity of measurements or tests, and should be part of an overall quality strategy. The main objective of a PT scheme is to help the participant to assess the accuracy [12] of its measurements. In addition, participation in an appropriate PT scheme is required for laboratories seeking recognition of their competence through accreditation against the standard ISO/IEC 17025 [13] or ISO 15189 [14]. In some sectors participation in specific schemes can be mandatory [15]. PT schemes are operated for the benefit of the laboratory participants. However, other parties also have a legitimate interest in PT schemes. These include customers of analytical laboratory services, accreditation bodies, regulatory authorities and other end-users of the laboratory results. The authors, Isabel Taverniers and all reported in the publication” Trends in quality in the analytical laboratory: Analytical method validation and quality assurance”, the different

approaches to validation, giving attention to the different characteristics of method performance with the concepts of single-laboratory, or in house validation, inter-laboratory or collaborative study [16]. The basic use of PT is to assess the performance for the conduct of specific measurements and calibrations.

This paper focused our experience on the inter-laboratory exercises undertaken for the evaluation of the analytical laboratory competency during the last decade in particular for the two new Proficiency Testing rounds organized by the IAEA under the RAF/4/022 project [17] in conjunction with WEPAL, the Wageningen Evaluating Programs for Analytical Laboratories ([www.wepal.nl](http://www.wepal.nl)). [18]

## II. SYNOPSIS COMPETENCY TESTS PARTICIPATION OF THE NAA-CRNB LABORATORY

Our NAA research laboratory was participated in several inter-comparison tests organized in the frame work of AFRA Research Reactor Projects. In the first participations, the work of analysis is based on the inter-comparison results of African laboratories. The last PT testing participation was to evaluate our research laboratory among a great number reached 500 laboratories on an international scale. This contribution was carried out in the PT program launched by WEPAL organisation. In this context, a brief description was presented for all participation. Final Stage

### A. The AFRA IV-7 proficiency test 2003 using CMIP-P1 Rock Materials

Our first participation using NAA laboratory on the proficiency test was carried out in the frame work of the AFRAIV-7 project named CMIP-AFRA-P1 Test 2003. It was requested from the laboratories to determine the trace and minor elements in geological materials. The proficiency test study was organized by the CSIR-NML in collaboration with IAEA and NECSA for the laboratories of the Research Reactor Centres in Africa. About 11 laboratories from 5 countries were participated in this study for the determination of 16 trace and minor elements (Au, Ca, Cd, Co, Cr, Cs, Fe, K, Mn, Na, Ni, Pb, Rb, Th, U, Zn) in a rock-type test material. The analytical methods used in this study are INAA, EDXRF, GFAAS, IC and ICP-MS. This work aimed to illustrate the comparability of results produced by the participating laboratories [19]. The results of the proficiency testing exercises were evaluated by Experts to the IAEA and a follow-up Inter-comparison feedback Meeting was held in Algeria during 29 Nov-3 Dec 2003 [20].

### B. The AFRA IV-7 Proficiency Test 2005 using medicinal plants and aquatic-biomonitoring Materials

Two years later, AFRA IV-7 (2005) was the second Proficiency Test within the AFRA IV-7 Research Reactor Project for Socio-economic development. Thirteen laboratories from eight countries were participated in this study. The laboratories were requested to analysis 31 elements in five biological materials that were distributed by the project. One of the distributed samples was an IAEA

reference material (IAEA-155, Whey powder) to check the results of the nuclear analytical technique applied for all five materials. The focus of this project was on the evaluation of foodstuffs for their nutritive values, the determination of the element composition of medicinal plants and the analysis of aquatic-biomonitoring for environmental monitoring, using nuclear and related analytical techniques, with neutron activation analysis as the common technique [21]. The results of the proficiency testing exercises were evaluated by Experts to the IAEA and a follow-up Inter-comparison feedback Meeting was held in Libya during 2005 [22].

### C. The AFRA IV-12 Proficiency Test 2007 using foodstuff, medicinal plants and aquatic biomonitoring Materials

The AFRAIV-12 Inter-laboratory Analysis Test 2007 was the third inter-laboratory analysis test within that project. The laboratories were asked to analyze about 43 essential and toxic elements using nuclear and related analytical techniques, with neutron activation analysis as the common technique. Five materials were distributed to the participants representing foodstuff, medicinal plants and aquatic biomonitoring according to the focus of the RAF/4/020 AFRA IV-12 (2006–2010) project [23].

For the first time, Es-Salam research reactor of Algeria had the opportunity to take part with two techniques INAA and  $k_0$ -INAA for which the  $k_0$ -NAA technique was newly developed (2004) in our department. Indeed, two sets of independent results obtained by INAA and  $k_0$ -INAA techniques were sent for evaluation. Each technique was used as an analytical tool regarded as an independent laboratory while associating a number and a particular code in event the Algerian laboratories with CRNB/INAA and CRNB/ $k_0$ -NAA, respectively. The participating laboratories met to discuss their results in the presence of the experts of IAEA. All results obtained by the African analytical laboratories were evaluated by experts of the IAEA and the meeting for was held in Kenitra, Morocco from 14 to 18 July 2008 to discuss the results evaluations [24, 25].

### D. WEPAL PT scheme 2011-2012 rounds

The goal of this paper focuses the assessment of our scientific activities through the PT evaluation of the NAA laboratory. The principal actions carried out within the framework of the RAF/4/022 project: 'Enhancement of Research Reactor Utilization and Safety' led to the evaluation of the African laboratories with the advantage to participate on the PT program of WEPAL. The Proficiency Testing rounds related to the determination of (trace) elements in soil and plant materials were held by WEPAL for which 4 ISE and 4 IPE samples have been used in each round during 2011/4 and 2012/1, respectively. WEPAL, a provider of such Inter-laboratory comparison schemes, is accredited by the Dutch Council for Accreditation, for compliance with the International Standard ISO17043:2010. The activity within IAEA AFRA RAF 4/022 coincided with similar

assessments amongst European and Latin American analytical laboratories under RER 1007 and RLA 0037, respectively.

In this study, we aim to evaluate of the competency of our laboratory among the proficiency test 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.

Indeed, the IAEA facilitated during 2011-2012 laboratories, participating in IAEA/AFRA project RAF/4/022 to assess their analytical performance by taking part in analytical proficiency testing IAEA in conjunction with WEPAL, the Wageningen Evaluating Programs for Analytical Laboratories.

Two such rounds have been organized, and the results evaluated by the IAEA supported by Experts in nuclear and non-nuclear analytical techniques. Potential sources of error, technical and organizational, were identified for the individual laboratories, action plans for improvement were drafted and accepted, and the Agency was recommended continuing this approach for proficiency testing and evaluation as all participants highly valued the outcome of it. The results of analysis have been reported according to the specific forms for IPE and ISE reports. During the meeting on Inter-Comparison Feedback of NAA and other Analytical Techniques Proficiency Tests, AIEA Project/RAF4/022 - Enhancing Research Reactor Utilization and Safety, 04-08 June 2012, Tunis, Tunisia, all participating laboratories have been presented their results with the same IAEA proposed form.

#### *E. WEPAL PT scheme 2013.1 round*

Laboratories, participating in IAEA/AFRA RAF 4/022, IAEA/ARCAL RAL 0037 and IAEA RER 4/032 - RER 1/007 participated in the year 2013, as well as in previous years 2010, 2011, and 2012 in consecutive interlaboratory comparison rounds organized by the Wageningen Evaluating Programs for Analytical Laboratories (WEPAL). Four soil samples and 4 botanical (plant) samples were provided in each round for analysis. The IAEA has, together with participants, evaluated the results from the African laboratories and European laboratories in feedback workshops to discuss sources of error and provide recommendations for improvement. During the meeting on Inter-Comparison Feedback of NAA and other Analytical Techniques Proficiency Tests, AIEA Project/RAF4/022 - Enhancing Research Reactor Utilization and Safety, 27 -32 May 2013, Vienna- Austria. All participating laboratories have been presented their results with the same IAEA proposed form [26].

### III. EXPERIMENTAL

#### *A. Materials analyzed*

The global program of WEPAL included six sets of samples: ISE (International Soil-Analytical Exchange), IPE (International Plant-Analytical Exchange), SETOC (International Sediment Exchange for Tests on Organic Contaminants), MARSEP (International Manure and Refuse sample Exchange Program), BIMEP (International Biomass Exchange Program) and Q (Quasimeme Laboratory Performance Studies organic contaminants, metals, nutrients in seawater, sediment and biota).

Each quarter, sample kits are mailed to participating laboratories and data are analyzed to evaluate performance. Each mailing and subsequent data analysis is referred to as a round. The reference samples included in a kit are designated to cover a variety of analytical methods used by laboratories. In the case of the AFRA IV-12-RAF4/022 project within the financial support of IAEA a total of sixteen materials were received from WEPAL organism during 2013.1 periods.

The materials representing different types of soil and plant named ISE and IPE. In each round the samples ISE and IPE are labelled from one to four. The soil and plant materials are sandy soil, calcareous brown soil, clay, river clay, clay soil, loamy soil sandy clay soil, Leek (allium porrum), Lucerne (Medicago Savitum), Oil palm leaves (Elaeis Guineensis), Grass gr94 (Poaceae), Maize (Zea mays), Banana leaves (Musa Sapientum) and Beech Leaf (Fugus Sylvatica). Before receiving the WEPAL materials, all samples used in these rounds of the proficiency test have passed the homogeneity test [27-30].

#### *B. The $k_0$ -standardization based neutron activation analysis $k_0$ -INAA*

The  $k_0$ -based neutron activation analysis ( $k_0$ -NAA) technique, developed in 1970s, is being increasingly used for multielement analysis in a variety of matrices using reactor neutrons. In the  $k_0$ -INAA the evaluation of the analytical result is based on the so-called  $k_0$ - factors that are associated with each gamma-line in the gamma-spectrum of the activated sample. These factors replace nuclear constants, such as cross sections and gamma-emission probabilities, and are determined in specialized NAA laboratories. This technique has been reported to be flexible with respect to changes in irradiation and measuring conditions, to be simpler than the relative comparator technique in terms of experiments but involves more complex formulae and calculations, and to eliminate the need for using multi-element standards. The concentration of an element can be determined as:

$$\rho_x (\text{ppm}) = \frac{\left[ \frac{N_p/t_m}{\text{SDCW}} \right]_x}{\left[ \frac{N_p/t_m}{\text{SDCW}} \right]_{\text{Au}}} \cdot \frac{1}{k_{0,\text{Au}(x)}} \cdot \frac{G_{h,\text{Au}}f + G_{e,\text{Au}}Q_{0,\text{Au}}(\alpha\alpha)}{G_{h,x}f + G_{e,x}Q_{0,x}(\alpha\alpha)} \cdot \frac{\varepsilon_{p,\text{Au}}}{\varepsilon_{p,x}} \times 10^6 \quad (1)$$

where the indices  $x$  and  $\text{Au}$  refer to the sample and the monitor, respectively;  $W_{\text{Au}}$  and  $W_x$  represent the mass of the gold monitor and the sample (in g);  $N_p$  is the measured peak area, corrected for dead time and true coincidence;  $S$ ,  $D$ ,  $C$  are the saturation, decay and counting factors, respectively;  $t_m$  is the measuring time;  $G_{th}$  and  $G_{epi}$  are the self shielding correction for the thermal and epithermal neutrons respectively.

C. Procedure

Triplicates were prepared from each sample of IPE and ISE. To be able to exploit both techniques INAA and  $k_0$ -INAA in the same experiment, it is necessary to prepare the CRMs, RMs and gold monitors. In this work, the certified reference materials like CRM-GSV4, CRM-NIST-1573a, CRM-GSD12 and reference material such as RM-AIEA-soil7 were used to determine the elemental concentrations by relative technique. In the other hand, the analytical results obtained of CRMs are the subject of internal quality control procedure.

All samples, monitors and standards were placed in clean cylindrical aluminium vials for long irradiation. The gold monitors Al-0.1% Au alloy wire of 0.5 mm diameter provided by IRMM (Institute for Reference Materials and

Measurements, Belgium) were prepared for  $k_0$ -INAA utilisation. Data acquisition is performed using Genie-2k software from Canberra [31]. All spectra are deconvoluted with the commercial software HyperLab 2005 [32] with resulting to files peak table (\*.PTF) and a spectrum (\*.SPE) which are used as input for KayWare software [33]. This is a specific software package developed for  $k_0$ -based NAA at DSM Resolve in the 1990s and at present commercialized by INW Gent and  $k_0$ -Ware in the Netherlands.

IV. RESULTS AND DISCUSSION

The PT program is designated to complete, not replace, a participating laboratory’s internal quality control (QC) program. In addition, the PT program may identify analytical problems when the laboratory’s internal controls do not submit an application. To get reproducible results, Wepal recommends to the participants the determination of the moisture content in each sample. The procedure is explained in the quarterly IPE-ISE reports of WEPAL for which the materials must be drying during at least 3 hours at 105 °C and cooling down in desiccator before weighing. The dry mass factors were determined for all plant and soil WEPAL samples. In our analytical techniques, the moisture content is directly introduced as an input values in the KayWin program.

The results are reported for the RAF/4/022 inter-laboratory proficiency test 2013 and consequently evaluated by WEPAL. The final treatment of results for each type of samples ISE and IPE has to generate some parameter statistics such as: mean, standard deviation, median, MAD and Z-score.

Table 1: Experimental conditions used in this work

Parameters	Irradiation for geological samples	Irradiation for biological samples
WEPAL Materials	ISE	IPE
CRM and RM	CRM-GSD12 Soil7	GSV4 Lichen336
Neutron spectrum parameters ( $\alpha$ , $f$ , $\Phi_{th}$ )	$\alpha = 0.027$ , $f = 28.83$ , $\Phi_{th} = 4.7 \cdot 10^{+12}$ [2]	$\alpha = 0.045$ , $f = 20.5$ , $\Phi_{th} = 3.76 \cdot 10^{+13}$ [1]
Irradiation time	4H	8H
Decay time	4-6 d 11-12 d	6 – 8 d 12 – 16 d
Counting time	5400 sec 7200 sec	5400 sec 7200 sec
Measured element	As, Ba, Br, Ca, Cd, Ce, Co, Cr, Cs, Fe, Ga, K, La, Mo, Na, Nd, Rb, Sb, Sc, Sr, Th, U, W, Zn, Zr	As, Ba, Br, Ca, Co, Cr, Cs, Fe, K, Mg, Mn, Na, Rb, Sb, Se, Sr, Zn

The assessment of the PT rounds participation during 2013 has been evaluated according to the sophisticated statistical methods which are required to obtain meaningful assessments. The model that is chosen calculates population characteristics (mean and standard deviation) from experimental datasets. The model uses an estimate for the

probability density function of the measurement process and calculates a best fit based on all observed values. The analytical results of the analyzed materials are presented by three ways per element, sample and participant (laboratory) in the quarterly and annual reports ISE and IPE of WEPAL. In this study, the evaluated results obtained by our laboratory were carried out by WEPAL for which the proficiency tests

of the participant laboratories were processed by the tool of Z-scores. Using this approach, all Z-score values of our laboratory were presented graphically in the figures 1, 2, 3

and 4. According to the evaluation processed by the PT provider the majority of our results correspond to Z-scores lower than 2.

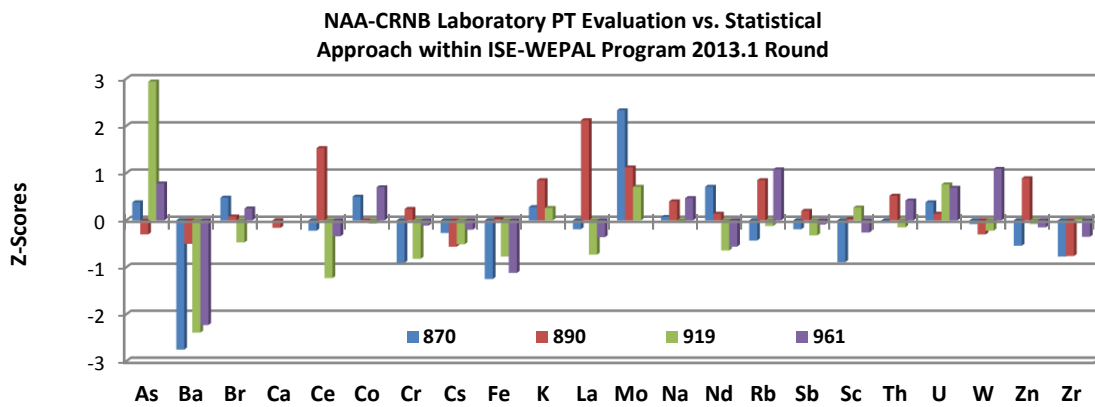


Fig 1: Graphical PT evaluation of the overall z-scores results obtained by NAA-CRNB laboratory 22 elements analyzed in four ISE WEPAL materials.

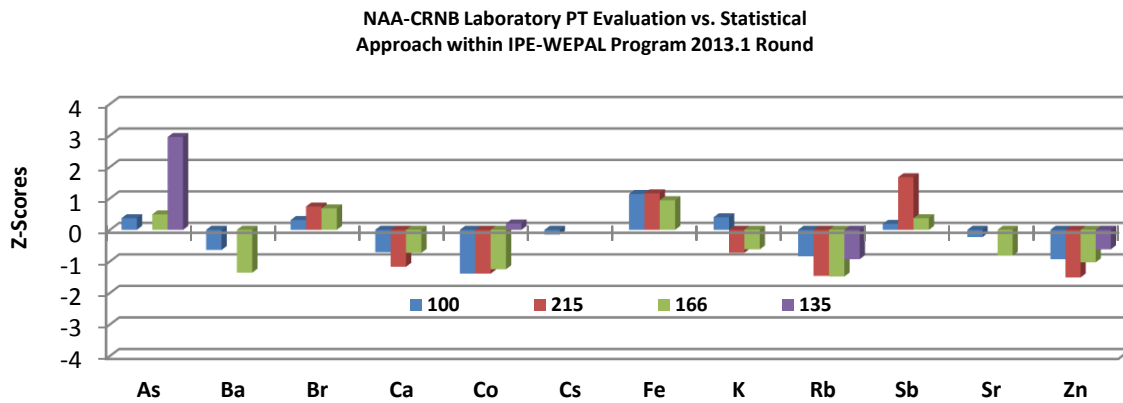


Fig. 2: Graphical PT evaluation of the overall Z-scores results obtained by NAA-CRNB laboratory 12 elements analyzed in four IPE WEPAL materials.

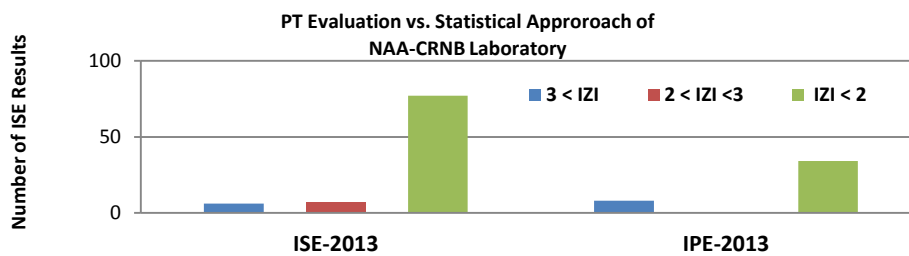


Fig. 3: Proficiency test assessment of the NAA-Laboratory results obtained for 2013.1 round

The success rate became more important during our second participation in PT of provider. This indicates that the system calibration like calibration of weighing balance, irradiation channels and HPGe detectors were obtained carefully. In a short-term projection our laboratory seeking accreditation, these measures are an important aspect of the requirements. PT is one of these measures, as well as for example, the use of (certified) reference materials. PT plays a highly valuable role as it provides an objective evidence of the competence of the participant. This evidence can be used to improve the

performance of the participant and/or give confidence in the participant's ability to perform a specific measurement.

During the participation in PT schemes, it is very interesting to assess the quality of obtained results and to make comparison at different stage. In the figure 5, the comparison was presented. The PT round 2013.1 revealed that the number of satisfactory results expressed by accepted Z-scores are very important. In addition, an apparent improvement was observed for the second test. This indicates

the role of researcher in the improvement of the analytical process.

#### V. CONCLUSION

One of the basic elements in all PT is the evaluation of the performance of each participant. In order to do so, the PT provider has to establish two values, which are used for the performance evaluation. It is clear that the PT plays a highly valuable role as it provides an objective evidence of the competence of the participant. This evidence can be used to improve the performance of the participant and/or give confidence in the participant's ability to perform a specific measurement.

This paper presents the general state of the NAA analytical laboratory with the considerable efforts of NAA group using Es-Salam research reactor facility. During the last decade, we acquired a good experiment through our participations in the proficiency tests program and the inter-comparison exercises which were carried out within the framework of the AFRA/RAF projects. In particular, we agreed that the objectives of the IAEA in facilitating these PT rounds under the project RAF/4/022 were achieved. These remains strongly recommend that the IAEA continues to facilitate new PT Rounds with feedback meetings for independent assessment of performance of analytical laboratories. In addition, the first participation with WEPAL organization we got a clear understanding of the procedures and methodology of PT rounds, and a mechanism for their own evaluation of the outcome thereof.

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