ESTABLISHMENT OF ADVANCED QUALITY SYSTEM FOR NUCLEAR ANALYTICAL LABORATORIES

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Abstract
Comprehensive Quality Control (QC) and Quality Assurance (QA) Program is stated on the quality policy, organization, methods and records for nuclear analytical laboratories which are necessary for improvement of productivity, to upgrade the performance, credibility and reputation. The proper and complete identification of quality elements for management and technical requirements are being written in Quality Manual as well as analytical and organizational procedures and working instructions according to ISO 17025 standard. Technical ability of γ , X-ray and α/β laboratories in Center is being checked by participation in proficiency test, critical technical variables, and quality results. Performance of quality system is being controlled by external audit inspection, progress reports and service to clients.Present study is a framework of model project of IAEA, coded RER/2/004, which is resulted self-sustainable accreditation from national body, TURKAK.

Introduction

Quality Assurance is defined as planned activities that are designed to ensure that the quality control activities are being properly implemented. The quality assurance program is a monitoring and auditing program that is designed to examine overall laboratory performance, evaluate it in detail, and disclose the cause of any less than satisfactory conditions. A successful quality system will lead directly to procedures that need changing, equipment that requires upgrading analysts who are in need of training or better supervision, and so forth. This need has lead to a proliferation of so-called accredition programs that are being widely used to certify laboratories.[1] The International Standard, ISO 17025, has been produced as the result of extensive experience in the implementation of ISO/IEC Guide 25:1990 and EN 45001:1989 both of which it now replaces. It contains all of the requirements that testing and calibration laboratories have to meet if they wish to demostrate that they operate a quality system are technically competent, and are able to generate technically valid results. The use of ISO 17025 should facilitate cooperation between laboratories and other bodies to assist in the exchange of information and experience, and in the harmonization of standard and procedures.[2]

The regional technical cooperation project of IAEA as RER/2/004 is a model-pilot project and had been approved in 1999 for a period of two years aiming at the implementation of a comprehensive QA/QC protocol in laboratories of Member States following the ISO guide 17025. 13 Nuclear Analytical Laboratories (NALs) are selected from countries of Central and Europe and the Newly Independent States, such as Turkey, Slovenia, Slovakia, Romania, Poland, Latvia, Hungary, Estonia, Croatia, Belarus, Armenia and Agency's laboratory in Seibersdorf.

This very demanding project required the submission of 6 monthly progress reports, two external audit inspections and participation in two proficiency test rounds. Progress of the individual laboratories was monitored by a concise scoring system that was applied to the submitted progress reports and the audit reviews.[3] Nuclear Analytical Laboratories (gamma, X-rays and alpha/beta) of Ankara Nuclear Research and Training Center were selected by IAEA as participant with appropriate staff and facilities available for full participation in the model project.

The NALs in Ankara made reasonable progress in the implementation of its QA system, a concise system for documentation, establishment of standard operating procedures, procedures for validation of methods, surveillance of method performance, systems for sample management, regular qualification of personnel client liaison and safety during the two year span of the RER project. The results of Progress reports and external audit inspection reports are quite good. PT sample results are compatible.

Quality Assurance Principles

Establishment and improvement of QA/QC of nuclear analytical techniques was based on quality indicators of Agency according to the ISO 17025. International Standard of ISO 17025 specifies the technical and management requirements for the competence to carry out tests and/or calibrations, including sampling. It covers testing and calibration performance using standard methods, non-standard methods and laboratory-developed method.[2] Table 1. shows some of quality indicator of IAEA for quality system of NALs.The proper objectives of the quality assurance program were clearly stated and understood by both management and project staff. The following are a few objectives of our quality assurance system: [3]

- To upgrade the quality of gamma, alpha/beta and X-ray laboratories performance
- To maintain a contiuning assesment of the quality of data generated by staff
- To provide permanent record of instrument performance in NAL's a basis for validating data and projecting repairs and replacement needs
- To ensure sample integrity

- To improve record keeping
- To improve of productivity
- Establishment of the NALs credibility
- Good reputation and good position on the market
- More grants, more services, more customers.

From the very beginning, the entire laboratories staff were involved in planning and developing the quality assurance program, with consensus approval obtained at various steps from key affected personnel.Project staff including management established policy, assigned responsibilities, set standards, provided technical expertise, technical advice and guidance, and did the actual writing and reviewing of assigned parts of the plan.

The layout and the quality indicators for analytical and organizational requirements are assessed separately and the program on QA/QC was perfectly applied. Some achievements, corresponding the direct benefit of gamma, alpha/beta and X-rays are summarized as below:[3]

- Management of the information
- Documented procedures
- Better organization of the laboratories
- Preparation of new normative documents
- Drafting-preparing quality manuals
- Team work, higher motivation of the staff
- Uncertainty budget implementation
- Management of the nonconforming work
- Improved trackability
- Increased confidence in work
- Definition of performance criteria

Experimental

The following techniques were selected in participation of project

- Alpha and beta counting
- γ -ray spectrometry
- X-ray fluorescence spectrometry

Thirteen NALs staff members were fully committed to the project. Most routine analytical methods were based on established standard methods. All routine methods were thoroughly validated authorized before use in laboratory. Flowchart of process control of nuclear analytical laboratories in Center is given in Figure 1. Methods and projects reviewed annualy. Quality Control samples were analyzed with each batch of samples to ensure the validity of the analytical results.Control standards, blanks, standard reference materials were employed as quality control protocols. Repeatibility is one of the measurement of method validation and its requirements are same sample, analyst, apparatus and laboratory. As an example,

Figure 2 shows repeatability measurements of X-ray fluorescence techniques by using ZnKα of MES standard for ED2000 spectrometer.

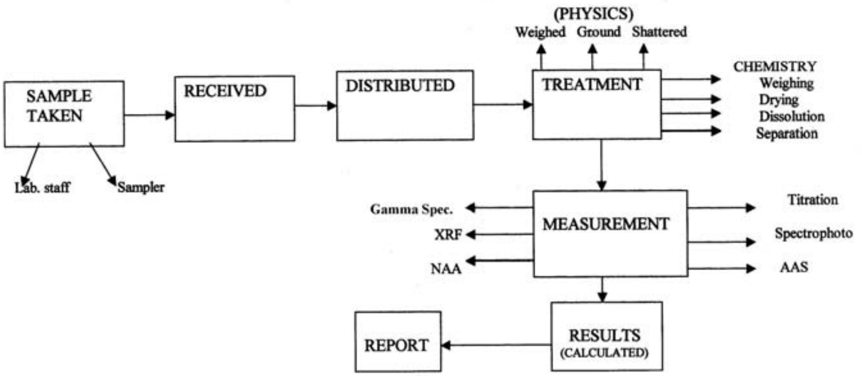

Figure 1. Process Control Flowchart of NALs

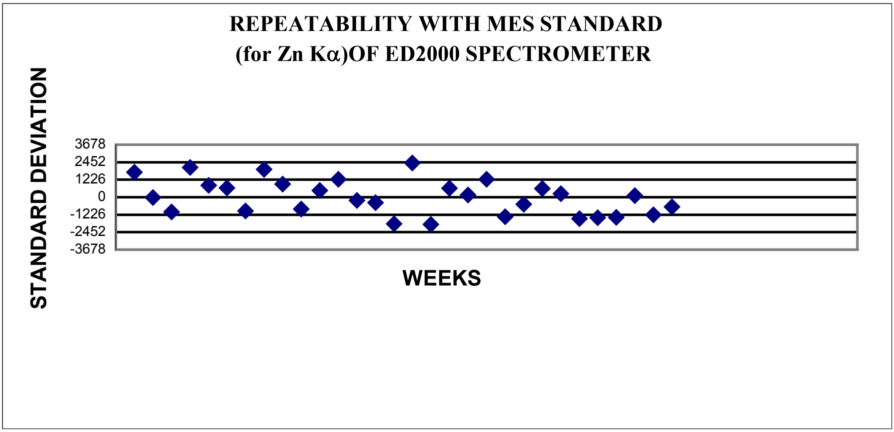

Figure 2. Repeatability results of X-ray fluorescence spectrometry

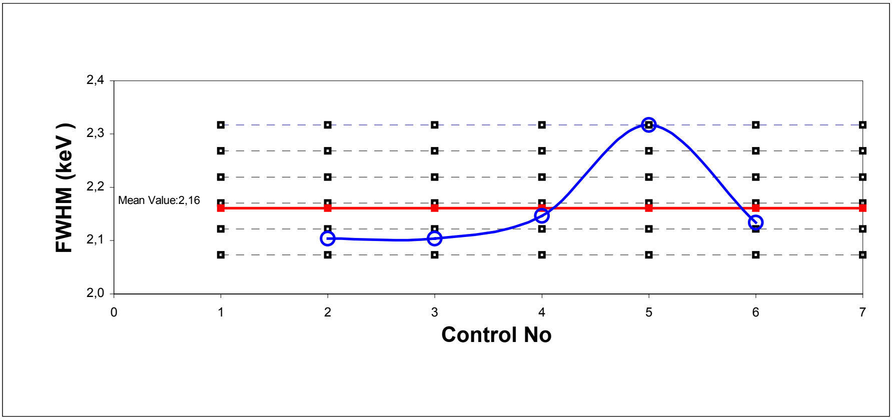

Figure 3. FWHM control chart at 1333 keV -γ energy for tube geometry.

A useful tool in the quality assurance program is control charting. A control chart is a graphical plot of test results with respect to sequence of measurements and in a state of statistical control (FWHM,background, humidity, temperature, quality control results...). As an example, FWHM control chart at 1333 keV- γ energy of Co-60 source for tube geometry in γ laboratory is given Figure 3.

Analysis of Proficiency Test Samples

A frame of this project, to evaluate the participants performance in the determination of selected radionuclides of environmental importance and to validate the accuracy and precision of measurements, two proficiency tests were carried out. Radionuclides in spiked soil sample and in standard solution were determined.

IAEA Quality Control Service has sent to α/β laboratory spiked soil samples for determination of ^{90}Sr activities in intercomparison run program. Samples were analyzed according to the IAEA Technical Reports Series No.295.[4] Activity of ^{90}Sr in samples were calculated by

$$A = \frac{\mathbf{R}_2 \mathbf{f}}{(\%\mathbf{Sr})(\%\mathbf{Y})\mathbf{E}\,\mathbf{M}\,\mathbf{e}^{-\lambda t}} \qquad (1)$$

Combined standard uncertainty was calculated by

$$\mathbf{S} = \frac{(\mathbf{Netcount(cps)} \pm 1\sigma)\,\mathrm{f}}{(\%\mathbf{Sr} \pm 1\sigma)(\%\mathrm{Y} \pm 1\sigma)(\%\mathrm{E} \pm 1\sigma)(\mathrm{M} \pm 1\sigma)} \qquad (2)$$

where,

R_2 : Net count
%Sr : Chemical yield of Sr
%Y: Chemical yield of Y
E: Counting efficiency for ^{90}Y
t: time between Sr precipitation and middle of Sr measurement
M: Sample mass
f: Correction factor

The comparison between analysis results and IAEA data is shown in Table 2. Accuracy and precision criteria are given in Table 3. IAEA has applied statistical treatment using u-test for the evaluation of all results.

Table 1. Some of quality indicators

Management commitment	Calibration
Mission statement	Trackability
Critical technical variables	Documentation control
Environmental factors	Traceability
Laboratory management	Method Validation
Uncertainty budget	Sample Custody
Internal audits	Quality Manual
Statistical analysis	SOP

Table 2. Comparison of analysis results on Sr-90 and reference values.

Sample Code	Unit	IAEA Data		Reported Results		Relative bias, %	z-Score	u-test score
		Value	Uncertainty	Value	Uncertainty			
RER/2/004/TU-ANK/3	Bq	0.1718	0.00073	0.11	0.02	-36.0	-1.80	3.09
RER/2/004/TU-ANK/4	Bq	0.1718	0.00073	0.09	0.02	-47.6	-2.38	4.09
RER/2/004/TU-ANK/5	Bq	0.1718	0.00073	0.13	0.02	-24.3	-1.22	2.09
RER/2/004/TU-ANK/6	Bq/g	-	-	0.004	0.0010	-	-	-
RER/2/004/TU-ANK/7	Bq/g	21.81	0.077	22.05	0.07	+1.10	+6.99	2.31

Table 3. Accuracy and precision criteria for Sr-90

Sample Code	Accuracy criteria			Precision criteria		Final Status
	$\lvert$Value $_{IAEA}$- -Value $_{Analyst}\rvert$	$3.29\sqrt{Unc^2_{IAEA}+Unc^2_{Analyst}}$	Status	%	Status	
TU-ANK/3	0.062	0.066	passed	18.2	passed	passed
TU-ANK/4	0.082	0.066	failed	22.2	passed	rejected
TU-ANK/5	0.042	0.066	passed	15.4	passed	passed
TU-ANK/6	-	-	-	-	-	-
TU-ANK/7	0.240	0.34	passed	0.5	passed	passed

In addition, gamma-emitting radionuclides in the spiked soil samples and standard solutions prepared by IAEA for proficiency tests have been analyzed by using a gamma-ray spectrometer with n-type detector. The full energy detection efficiency as a function of gamma-ray energy was determined by radioactive standard containing ^{109}Cd, ^{57}Co, ^{123}Te, ^{51}Cr, ^{113}Sn, ^{137}Cs,^{60}Co and ^{88}Y radionuclides. For a given source-sample geometry, two weighed spiked soil samples and two standard solutions were counted for the counting periods of 100000s, 150000s and 235000s depending on the level of activity of the sample to ensure good statistical quality of data. The specific activity of any radionuclide in the sample using net counts of the peak of interest in the measured spectrum was calculated the following formula.

$$\mathbf{A}=\frac{\mathbf{N_p}/\mathbf{t_c}}{\mathbf{f_\gamma E_p(E_\gamma)D}}\times\frac{1000}{\mathbf{w}} \tag{3}$$

Where A is the specific activity Bq. Kg^{-1}, w is sample weight(g), fγ isgamma emission probability per decay nuclide, $E_p(E_\gamma)$ is full energy peak efficieny for a given E_γ energy, D is decay correction factor (exp(-0,693t/$T_{1/2}$) in which t is decay time from reference date to start of MCA and $T_{1/2}$ radionuclie half life the lowest detectable activity level(MDA) in Bq.g^{-1} for the peak of interest at the 90% confidence level is calculated as follows

$$\mathbf{MDA}=\frac{3\sqrt{\mathbf{N_{bkg}}}}{\mathbf{t_c\varepsilon_p(E_\gamma)f\gamma w}} \tag{4}$$

where N_{bkg} is the background count collected during time t_c for the peak of interest. As an example, he measured results and IAEA data for spiked soil sample is given in Table 4. IAEA treatment for all results are given in Table 5. It is seen from Table 4-5 that most of the measured results are consistent with the target values(i.e, IAEA data). The uncertainty sources for activity calculation are: 1) the accuracy of the gamma emission yields and isotope half-lives taken from NUDAT database[5] and Tuli's Wallet Card[6] 2) the accuracy of sample weigh determination (±0,1 mg balance accuracy) 3) the uncertainty in the peak area is expressed as the percent error:

$$\%\,\mathbf{error} = \frac{\mathbf{m\sigma_p}}{\mathbf{Np}} \times 100 \qquad (5)$$

where σ_p= the peak area uncertainty(standard deviation) N_p: the net area of the region of interest, and m=1,65 confidence level 4) the uncertainity of absolute efficiency (full-energy peak effiency) of dedector is determined from the calibration curve. They are order of ±3%. The final combined uncertainty for each radionuclide activity is calculated as the square root of the sum of variances of all above mentioned sources of the uncertainty.

Table 4.The results of radionuclides in spiked soil sample by gamma spectrometry

Radio-nuclide	Unit	IAEA Data		Reported Results		Relative bias, %	z-Score	u-test score
		Value	Uncertainty	Value	Uncertainty			
^{57}Co	Bq/g	37.8	0.92	36.84	0.82	-2.43	-0.16	0.74
^{60}Co	Bq/g	93.5	1.61	89.52	2.24	-4.26	-0.28	1.44
^{65}Zn	Bq/g	29.5	1.28	26.00	2.77	-11.7	-0.78	1.13
^{134}Cs	Bq/g	73.0	1.47	74.21	2.23	1.68	-0.11	0.46
^{137}Cs	Bq/g	84.2	1.79	86.55	1.71	2.80	-0.19	0.95
^{241}Am	Bq/g	93.1	7.5	82.86	1.39	-11.0	-0.73	1.34

Table 5. Accuracy and precision criteria for soil sample results.

Radio-nuclide	Accuracy Criteria			Precision Criteria		Final Status
	$\lvert Value_{IAEA} - Value_{Analyst} \rvert$	$3{,}29 * \sqrt{\mathbf{Unc}^2_{\mathbf{IAEA}} + \mathbf{Unc}^2_{\mathbf{Analyst}}}$	Status	%	Status	
^{57}Co	0.92	4.06	Passed	3.3	Passed	Passed
^{60}Co	3.98	9.08	Passed	3.0	Passed	Passed
^{65}Zn	3.45	10.0	Passed	11.5	Passed	Passed
^{134}Cs	1.23	8.79	Passed	3.6	Passed	Passed
^{137}Cs	2.35	8.15	Passed	2.9	Passed	Passed
^{241}Am	10.2	25.1	Passed	8.2	Passed	Passed

Conclusions

The progress we made that demonstrable and measurable improvement of the organizational and analytical performance of Nuclear Analytical Laboratories at Ankara Nuclear Research and Training Center in accordance to internationally harmonized requirements of ISO 17025 standard.With this project we had to evaluate our progress every half year and we had to send it in the prescribed form to the Agency. The results of five Progress Reports (quality indicators) and two audit inspection reports are given Table 6.

Table 6. Evaluation of Progress and Audit Reports

Progress Report No.	Score	Percentage	External Audit Inspection
0	30/108	27 %	
1	55/108	51%	48 %
2	68/108	63%	
3	86/108	80%	75%
4	100/108	93%	

Performance indicators show the increasing score from %48 to %75 for external audit inspections of our laboratories. On the other hand, biannual progress reports indicates improvement from %27 to %93 in two years. Proficiency test were used to evaluate the technical competence of the laboratories. Most of the our results are compatible.

Acknowledgement

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