QUALITY ASSURANCE AND QUALITY CONTROL IN NEUTRON ACTIVATION ANALYSIS: A GUIDE TO PRACTICAL APPROACHES

ANNEXES

CONTENTS

Annex I

BASIC PRINCIPLES OF CONTROL CHARTS

Control charts are one of the key tools in quality control (QC) and should be routinely used in the neutron activation analysis (NAA) laboratory. This annex introduces the basic concepts for the use of control charts in QC.

I–1. INTRODUCTION

The old saying "A picture tells more than a thousand words" may possibly have been in the mind of Walter Shewhart when he developed the concept of control charts in the early 1920s. A control chart is nothing less than presenting successive data in such a graphical way that interpretation in terms of acceptance/rejection is easier than by comparison of tabulated numbers.

There are two basically different types of control charts:

(a)   Control charts for presenting successive data that are not statistically correlated to one another. Examples are data of temperature, humidity, detector resolution (FWHM, Full-Width at Half-Maximum), peak centroid position etc.
(b)   (Shewhart) control charts for successive data that are statistically correlated to one another [I–1]. Examples are measured mass fractions of the same reference material, blank values, background counts, etc.

The interpretation of control charts is based on the testing of an hypothesis, e.g., an instrument performs well if the temperature is below a certain value, or that a measurement result is acceptable if it does not deviate more than a stipulated difference from a target value, or that successive results are statistically unlikely to differ from one another. Examples will be given below.

I–2. CONTROL CHARTS FOR NOT STATISTICALLY CORRELATED DATA

These control charts are used for presenting time series of recorded temperature, peak position, energy resolution. On the X-axis the date (and time, if relevant) is marked, and on the Y-axis the observed value. The Y-axis is also used to mark the boundary conditions for the variable, such as the maximum and minimum temperature. Normally these boundary conditions are denoted as the action or control limits, with upper and lower limits. Laboratories may also prefer to mark additional levels below the maximum, or above the minimum, where the variable is still within the acceptable range but where the operator should be alerted that a problem might be near. These are called the warning limits, and examples of these charts are shown in Fig. I–1.

Please note that for data charts that do not follow a normal distribution, it is not mandatory to have the upper and lower limits symmetrically around the reference or control value. In addition, there may be cases in which only a maximum level makes sense, such as for the detector's energy resolution. The energy resolution as measured by the manufacturer could serve as a minimum value, as it is unlikely that the laboratory will accomplish a better resolution, see Fig. I–2. In this case a value noticeably lower than the manufacturers' stated value could indicate a problem in the detector signal processing chain.
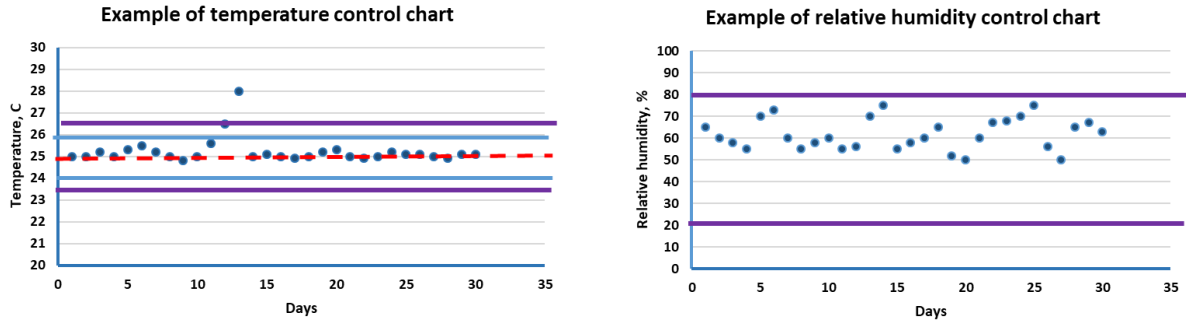
FIG I–1. Examples of control charts for temperature and relative humidity, showing control level (red, temperature only), action levels (purple) and warning levels (blue, for temperature only) (Courtesy of Mr. P. Vermaercke, SCK•CEN, Belgium).

Since the variation in FWHM values is not described by statistics, it does not make sense to calculate and mark the standard deviation of the data in the Shewhart control chart [I–2]. The charts allow for visual observation of trends or patterns, as shown schematically in Fig. I–2. It is however advised to perform a trend analysis (such as a regression analysis) and to draw a trend-line in order to detect a trend or a degradation of the HPGe detector performance for example due to vacuum problems that usually are very clear at high energies (see Fig. I–2 left) or sudden shift e.g. due to electronics problems that will show more obvious at lower energies (see Fig. I–2 right). In this case the warning and alarm levels are determined by the technical manager. For the FWHM, these are the levels that still can be used to perform sufficient spectrometry and that allow for separation of doublet and triplet peaks both at low and high energies.
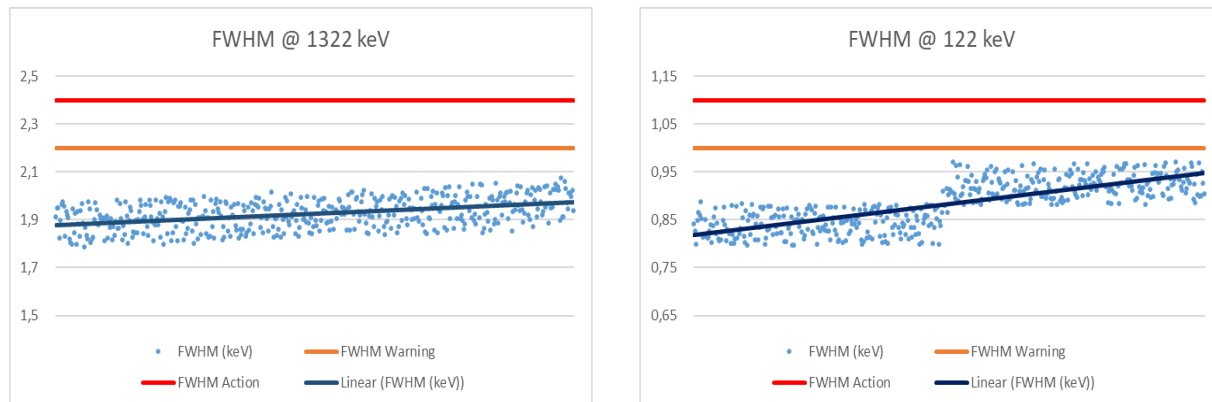

FIG I–2. Examples of patterns in successively recorded data as may show up in resolution control charts (Courtesy of Mr. P. Vermaercke, SCK•CEN, Belgium).

I–3. CONTROL CHARTS FOR STATISTICALLY CORRELATED DATA

The basic assumption in the normal Shewhart control chart is that successive data show a natural variation arising from the method of measurement, and that the values will be symmetrically distributed around a mean value in a normal or Gaussian distribution. Measurement values are plotted on the y axis against time of successive measurement on the x axis (which could be e.g. daily or weekly). Eventually, if sufficient data has been collected, the data histogram will start resembling the bell shape of the Gaussian distribution, see Fig. I–3.
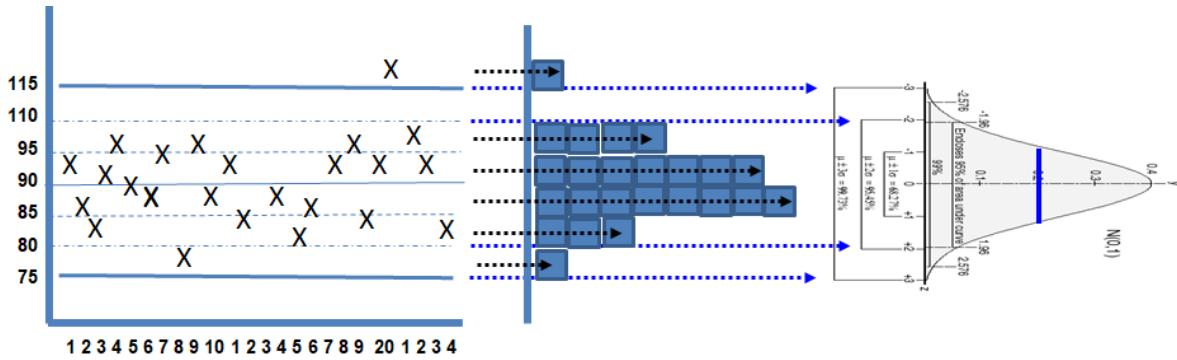
FIG I–3. How successive data lead to a histogram resembling the normal distribution (Courtesy of Mr. P. Bode, NUQAM Consultancy, Netherlands).

The distribution of values about the mean is governed by the standard deviation and statistically it is somewhat unlikely (5% probability) for a member of the population to be further away from the mean than two standard deviations, and very unlikely (0.3% probability) to be further away from the mean than three standard deviations. Thus 95% of measured results will always lie within ±2 standard deviations of the average, while 99.7% will always lie within ±3 standard deviations of the average.

The ±3 standard deviation levels are often considered the alarm limits, and the ±2 standard deviations are often considered as warning limits.

I–4. PITFALLS

The Shewhart control charts described here are built on the assumption that the data belong to a population with a normal distribution [I–3]. This is usually the case in radioanalytical work.

Several software packages are commercially available for converting measurement data into a control chart. Some of the commercial suites for gamma ray spectroscopy and NAA also have this option, referred to as 'quality assurance' or likewise.

It has been noted that some of the packages have a fundamental flaw, as the action and control limits are calculated from all data in the dataset, including all historical data. When this is done, the limits are thereby automatically adjusted once a new data point is entered, rather than the limits being kept fixed to verify an hypothesis. Consequently, all data in such charts will behave as being under statistical control even if gross outliers are present, as the limits will be adjusted to the value of such an outlier. An example is shown in Fig. I–4.
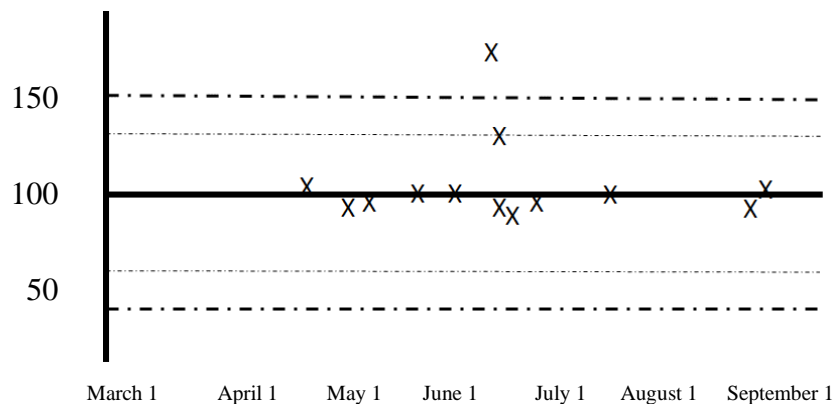
FIG I–4. Example of an automatically generated control chart in which the boundary limits are based on all data, including outliers. Note that the data by which the chart was started in April had a much smaller variation, but that the occurrence of two outliers around June 1 resulted in an increase of the standard deviation, whereas after adjustment, the variation is again at the same level (Courtesy of Mr. P. Bode, NUQAM Consultancy, Netherlands).

It is simple to verify the proper functioning of such software packages by deliberately adding a data point which is, e.g. a factor of 10–100 different from the central (mean) value, and observing if the boundary limits will change.

Another disadvantage of this method is that this leads to continuously updated warning and alarm limits which might be lower or higher than the previous value, but in the graph on the screen a fixed line is drawn for all historical data only based on the latest value. This means that a point that e.g. was within the 2 s warning level a month ago, might now show visually outside of the new 2 s warning level. Vice versa, a point outside the 2 s warning level might now show within the new 2 s warning level. This is confusing and may lead to undesired results. The preferred method is that warning and control levels are fixed from the beginning.

I–5. DEVELOPMENT OF SHEWHART CONTROL CHARTS

Control charts for non-statistical variables can be started by fixing the control and action limits at a convenient value (e.g. achievable control range of the counting room temperature), or based on experience (e.g. for detector resolution) or upon validation performance criteria [I–4]. Control charts with fixed warning and action levels are likely the most pragmatic and simple approach to be used in radio-analytical laboratories. For Peak position and efficiency checks, a multigamma source can be used covering low, medium and high energies. For the efficiency control chart, based upon a decay corrected activity of the nuclear source(s) used, one has to take into account the uncertainty on the calibration source, counting statistics and efficiency curve fitting (often high order polynomials are used). Overall, this leads to typical alarm limits in the order of 3–5%.

However, some users might prefer Shewhart (statistical) control charts that rely on the availability of information on the standard deviation of the population. For instance, if the mass fractions of elements in a certified reference material are plotted, the central value and standard deviation are known for the certified data, and the boundary limits are immediately known.
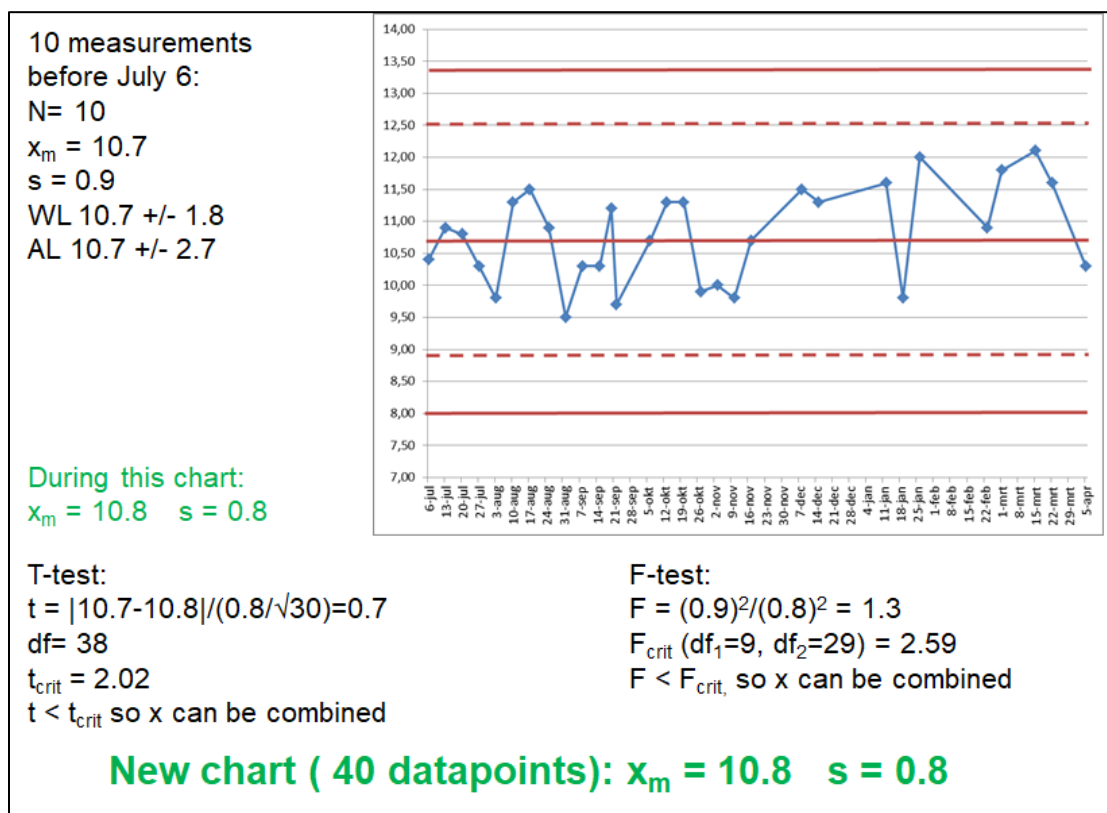
The figure contains the following text:

10 measurements
before July 6:
N= 10
x_m = 10.7
s = 0.9
WL 10.7 +/- 1.8
AL 10.7 +/- 2.7

During this chart:
x_m = 10.8   s = 0.8

T-test:
t = |10.7-10.8|/(0.8/√30)=0.7
df= 38
t_{crit} = 2.02
t < t_{crit} so x can be combined

F-test:
F = $(0.9)^2/(0.8)^2$ = 1.3
F_{crit} (df$_1$=9, df$_2$=29) = 2.59
F < F_{crit}, so x can be combined

New chart (40 datapoints): x_m = 10.8   s = 0.8

FIG I–5. *Worked-out example of an evaluation of a control chart. The chart's starting values have been based on the results of 10 measurements on July 6: 10.7 +/- 0.9. After 30 observations (September 5), the mean and standard deviation of the values are calculated (10.8+/- 0.8), and compared with a T-test and F-test with the starting values. As a result, it is concluded that the 30 data points belong to the same population as the 10 starting values; the data can be combined and form the basis of a new chart with slightly different mean value and boundary conditions (Courtesy of Mr. P. Bode, NUQAM Consultancy, Netherlands).*

The situation is different if the chart is used for plotting results of element mass fractions that are not certified and for which no standard deviation is given. In such a case, the laboratory should consider analysing the material 5–8 times under intermediate precision conditions (i.e. on different days, by different people, and differences in e.g. decay or counting time) and use both the mean value and the standard deviation of the measurement results.

However, both the mean value and the standard deviation are assumptions of the true values that have to be verified. To this end, the Shewhart control chart is evaluated after about 30 observations by a simple t-test — to verify if the mean of the 30 observations is in agreement with the starting value, and F-test — to verify if the 30 observations belong to the same population as assumed for which the starting standard deviation was used. A worked-out example is shown in Fig. I–5.

Such evaluations may result in continuous adjustment of the control and action limits, e.g., because the laboratory is continuously getting new indications of the probably true values of the element mass fraction and its standard deviation (see Fig. I–6). Please note again that this is not applicable to the Shewhart charts for results of certified elements since here the values are fixed.
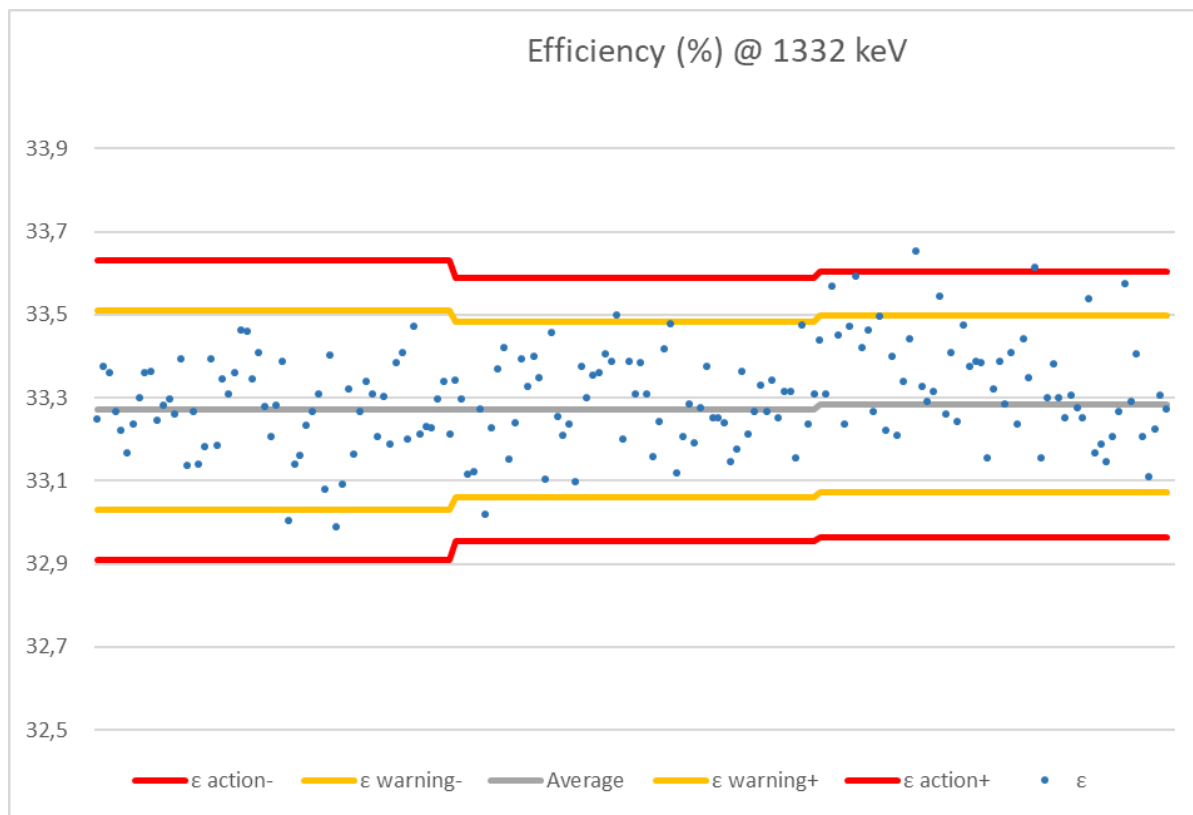
FIG I–6. Example of a control chart with adjustments following regular evaluations (in this example every 60 points) (Courtesy of Mr. P. Vermaercke, SCK•CEN, Belgium).

I–6. INTERPRETATION OF CONTROL CHARTS

Once the chart is set up, day to day results are plotted on the chart and monitored to detect unwanted patterns such as 'drift', or results lying outside the warning or action limits.

The evaluation of the charts is up to the professional expertise of the laboratory, and its risk assessment if data are outside the control limits or if obvious patterns are observed. There are recommendations for evaluation of Shewhart control charts based on probabilistic risk considerations [I–5, I–6]. These share the pitfall that a new data point triggers rather frequently a violation of one of the rules. As a consequence, a laboratory may face equally frequent non-conformity evaluations.

A laboratory should therefore justify its own evaluation criteria, based on risk assessment. It could be one of the conventions mentioned above, but simplified rules are often used, for instance:

1. Each violation of the 3 s level, or
2. Two consecutive violations of the 2 s level at the same side of the mean value $x_{m.}$

These are exemplified in Fig. I–7. Other rules do exist and can be applied with caution, such as the Westgard QC multirules [I–7].

A violation of acceptance criteria has to be followed by a careful examination. At large, it is recommended to implement and apply non-conformance procedures for this. It requires recording of the deviation, estimate of the root cause of the problem, inspection of its impact to the on-going and future work and, if relevant, an action to minimize the probability of recurrence (see NAA e-learning module Q6.5 [I–8] for more suggestions).
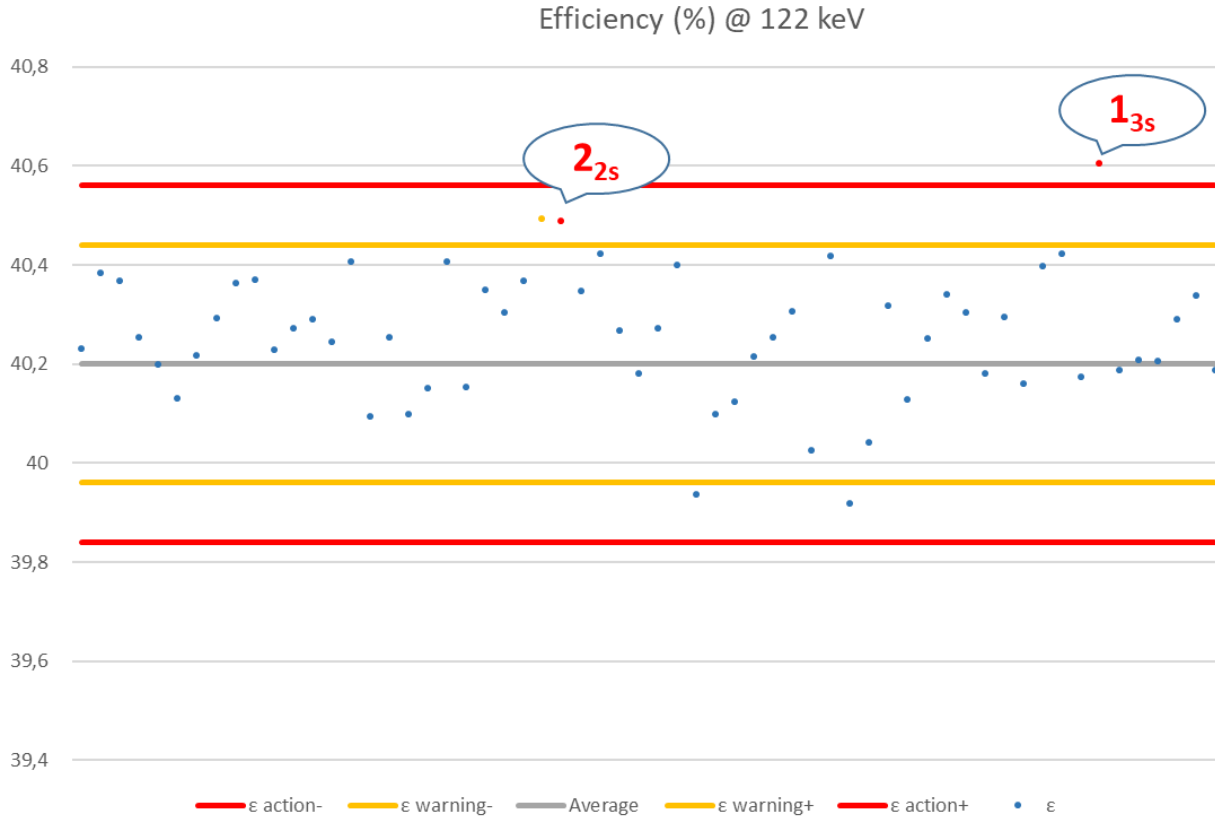

FIG I–7. Example of a control chart with a violation of the 3 s (1_{3s}) and with two consecutive violations of the 2 s level (2_{2s}) (Courtesy of Mr. P. Vermaercke, SCK•CEN, Belgium).

I–7. OTHER CHARTS

There are several other similar graphical ways to plot data for visual inspection, such as (but not limited to):

- X-bar chart. In this chart the sample means are plotted in order to control the mean value of a variable (e.g., size of piston rings, strength of materials, etc.).
- R chart. In this chart, the sample ranges are plotted in order to control the variability of a variable.
- S chart. In this chart, the sample standard deviations are plotted in order to control the variability of a variable.
- S^2 chart. In this chart, the sample variances are plotted in order to control the variability of a variable.
- CUSUM chart (Cumulative sum charts). CUSUM charts have been shown to be quite efficient in detecting small shifts (less than 2 standard deviations) in the mean of a process.

- C chart. In this chart, the number of defectives (per batch, per day, per machine, per 100 feet of pipe, etc.) is plotted. This chart assumes that defects of the quality attribute are rare, and the control limits in this chart are computed based on the Poisson distribution (distribution of rare events).
- u chart. In this chart the rate of defectives is plotted, that is, the number of defectives divided by the number of units inspected (the n; e.g., feet of pipe, number of batches). This chart does not require a constant number of units, and it can be used, for example, when the batches (samples) are of different sizes.
- Dispersion diagrams (Box-Whisker plots). These are used to display the main pattern in the distribution of data. The graph shows each value plotted as an individual point against a vertical scale. It shows the range of data and the distribution of each piece of data within that range.

As treatment of all these approaches is beyond the scope of this publication, the reader is referred to books on statistical QC and the internet for associated information, details and applications of such charts.

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Annex II

INHERENT QUALITY CONTROL OF NEUTRON ACTIVATION ANALYSIS — EXAMPLES

One of the advantages of NAA is its self-validating character due to the use of several dimensions such as intensity and energy and a third dimension that most analytical techniques cannot use, which is time, whereby decay is used as another possibility of inherent QC. Section 6.2 has further information on this subject. This annex presents several examples of the inherent QC of NAA, illustrating the concepts given in Section 6.2.

II–1. INTRODUCTION

Inherent QC of NAA refers to some of the inherent possibilities of QC that NAA offers in the framework of result validation. In NAA, as in other analytical techniques, one can use the dimension of energy, but additionally one can use the dimension of time and even different activation reactions.

Each of the examples given in this annex displays:

(a) The (n, γ) reaction used;
(b) The experimentally determined concentration for each gamma ray and measurements at different decay times (01, 02, 03, …);
— "Uncorr" refers to the uncorrected raw value (mass fraction) as calculated from the software
— "Corr" refers to the corrected value taking into account spectral, threshold or fission corrections [II–1]
(c) The calculated weighted average concentration for the element;
(d) The calculated expected weighted relative standard deviation of all results (based upon the experimental counting statistics for all peaks) in % denoted by "Expected StnDev %";
(e) The calculated observed weighted relative standard deviation for the end result (based upon the experimental counting statistics) in % denoted by "Observed StnDev %".

II–2. USING THE DIMENSION OF ENERGY: DETERMINATION OF Ag IN AN ORE SAMPLE

A measurement of an ore sample was carried out after a long decay time at close to end-cap position (about 1 cm) resulting in many coincidence effects (summing-out and summing-in) of the Ag-110m gamma ray peaks. Each Ag-110m peak, from 446.8 keV to 1562.3 keV was used for calculating the mass fraction of Ag — the most abundant peaks had counting statistics better than 1%. The mass fractions calculated using each of the peaks are given in Table II-1. The fact that the weighted average result has a very small observed weighted standard deviation (Observed StnDev %) of 0.5% validates:

(a) If (relative) instrumental NAA was applied: that no calculation errors were made (it is assumed that both sample and standard have the same geometry), that all spectral interferences for the chosen peaks are negligible or taken care of;

9

(b) If k_0-NAA was applied: the suitability of the chosen algorithm for coincidence effects and the experimentally determined efficiency calibration.

TABLE II–1. INHERENT QC – USING ENERGY: DETERMINATION OF Ag IN ORE SAMPLE [II–2]

Ag: Ag-109 -> Ag-110m	
Peaks (keV)	Mass fraction (mg/kg)
446.8	1524.8
620.4	1488.9
657.8	1527.9
677.6	1467.7
687.0	1524.2
706.7	1598.1
744.3	1587.7
763.9	1544.4
818.0	1495.4
884.7	1543.0
937.5	1538.3
1384.3	1578.0
1475.8	1572.0
1505.0	1616.7
1562.3	1618.6
Weighted Average	1543.2
Observed StnDev %	0.5%

II–3. USING THE DIMENSION OF ENERGY: DETERMINATION OF Se IN ORGANIC MATTER IN THE PRESENCE OF Hg

The determination of Se in the presence of Hg (or vice versa) is a difficult task in NAA. This is true both in relative instrumental NAA as the ratio of Se/Hg might be different in both sample and standard and in k_0-NAA, due to the spectral interference at 279.2 / 279.5 keV (if using Hg-203 and Se-75), the fact that both are long lived (and as such one cannot use the dimension of time) and that most other Se-75 gamma lines are also interfered in the spectrum by other radionuclides. The only way one can be certain that both the Se and the Hg values are reliable, in both the relative and k_0-NAA modes, is if all Se lines are coherent and as such serve as a QC for the quantification. Due to the low mass fractions of Se and Hg in organic matter it would be useful to use as many gamma lines as possible for the Se determination in order to improve the overall uncertainty. As an alternative, laboratories having a rabbit system might attempt to determine Se by Se-77m and using this value as a correction for the Hg determination or, if available, by using the Hg-value obtained from Hg-197m from the first measurement after one or a few days of decay.

Different measurements are needed: the first measurement (01) was performed after 4 days, measurement 02 was done after 9 days and measurement 03 was performed after one month. An iterative process is needed. It should be noted that in this example, counting statistics were about 5–10% depending on the gamma line for Se and 10% for Hg.

Starting from the interference-free 264.7 keV and 400.7 keV lines for Se-75, a first approximation for the mass fraction of Se can be made and a spectral interference correction for the contribution by Hg-203 on the 279.5 keV peak can be applied. This first approximation is then used in order to determine a better estimate for the Se mass fraction value at 279.5 keV, which then can be used to obtain a more robust average Se-value and one can start another iteration.

Normally only a few iterations are needed (see Table II-2, where it can be observed that in this example about 40% of the Se gamma line is due to the Hg-interference). The fact that when all spectral interference corrections are performed all five Se gamma lines (from 121.1 keV to 400.7 keV) yield an observed StnDev % well below the individual counting statistics and that the overall observed StnDev % is about 1.3% for all three measurements validates the chosen approach at different energies for Se, but at the same time validates the Hg- value:

(a) If (relative) instrumental NAA was applied: no calculation errors were made for the spectral interference correction even if the Se/Hg value is different in the standard and in the sample;
(b) If k_0-NAA was applied: the suitability of the chosen algorithm for spectral interference correction and for coincidence effects were validated, and also the experimentally determined efficiency calibration, as it is likely that the last Se-75 measurement is performed at close end-cap position.

TABLE II–2. INHERENT QC USING ENERGY: DETERMINATION OF Se IN ORGANIC MATTER [II–2]

Se: Se-74 -> Se-75		Mass fraction (mg/kg)					
	Peaks (keV)	01 (4 days decay)		02 (9 days decay)		03 (1 month decay)	
		Uncorr	Corr	Uncorr	Corr	Uncorr	Corr
	121.1	6.02	1.66	7.44	1.76	6.07	1.61
	136.0	1.89	1.79	2.01	1.86	2.02	1.89
	264.7	1.75	1.75	1.80	1.80	1.68	1.68
	279.5	2.92	1.65	2.99	1.74	3.06	1.84
	400.7	1.61	1.61	1.58	1.58	1.81	1.81
Weighted Average	1.77		1.73		1.79		1.78
Expected StnDev %	2.7%		5.1%		4.3%		5.0%
Observed StnDev %	1.3%		1.8%		2.0%		3.0%

II–4. USING THE DIMENSION OF TIME AND ENERGY: DETERMINATION OF Th AND K IN A SEDIMENT CRM

It is well known that the low energy peak of K-42 is interfered with a gamma line of Pa-233 at 312 keV. Two different measurements were carried out for the analysis of Th: one shortly (01, ~1 day) after irradiation and one after a long decay (02, ~1 month). In most cases the NAA operator will choose the second measurement for the determination of Th due to the half-life of Pa-233 of 27d. The first measurement clearly indicates interference from the short-lived K-42 result at 311.9 keV and would give a wrong result for Th at 311.9 keV (see Table II-3). Using the determined Th value, the 312.7 keV spectral inference on the K-42 peak can be corrected for. Coherence between the two K-42 gamma lines and the small observed standard deviation validates the K results (dimension of energy). The small observed standard deviation for Th for all gamma peaks in the 02 measurement (dimension of energy) validates that for the Th value, no calculation errors were made for relative instrumental NAA and validates the chosen approach for efficiency calibration for k_0-NAA. The fact that, after interference correction for the first measurement, at 312 keV both the 01 and the 02 measurement are coherent (dimension of time) also validates both the K and the Th results.

TABLE II–3. INHERENT QC USING TIME & ENERGY: DETERMINATION OF Th AND K IN A SEDIMENT CRM [II–2]

Th: Th-232 -> Pa-233	Mass fraction (mg/kg)				
	Peaks (keV)	01 (1 day)		02 (1 month)	
		Uncorr	Corr	Uncorr	Corr
	300.1	<9.545	-	8.30	8.30
	311.9	14.90	8.99	8.67	8.67
	340.5	-	-	8.49	8.49
	375.4	<87.192	-	8.46	8.46
	398.5	<44.295	-	8.90	8.42
	415.8	<36.774	-	9.43	9.43
Weighted Average	8.67		8.99		8.66
Expected StnDev %	0.4%		-		0.4%
Observed StnDev %	0.3%		-		0.4%
K: K-41 -> K-42	Mass fraction (mg/kg)				
	Peaks (keV)	01 (1 day)		02 (1 month)	
		Uncorr	Corr	Uncorr	Corr
	1 524.7	16 288.84	16 295.16	-	-
Weighted Average	16 295.16		16 295.16		-
Expected StnDev %	0.2%		0.2%		-
Observed StnDev %	0.5%		0.5%		-

II–5. USING THE DIMENSION OF TIME: DETERMINATION OF Gd AND Sm IN A SEDIMENT

The determination of Sm in a sediment or rock sample is difficult since the 103.2 keV gamma ray line of Sm-153 (half-life of 46.5 h) often suffers from interference with Gd-153, Pa-233 and Np-239 gamma lines, from respectively Gd, Th and U, and likewise the 103.2 keV line of Gd-153 (half-life of 240.4 d) suffers from interference with Sm-153, Pa-233 and Np-239 gamma lines. The fact that all four radionuclides have different half-lives helps to solve the problem. Measurement 01 is performed shortly (1 or 2 days) after irradiation, while measurement 02 was performed after three weeks to one month decay. Whereas the first measurement requires little correction for the determination of Sm due to mainly the U (Np-239) spectral interference, the second measurement requires a big correction mainly due to the Gd content (see Table II-4). The second measurement for the Gd determination requires mainly a correction for Th, while the first measurement requires a big correction due to the Sm and U interference. The fact that after correction both measurements at different decay times now agree validates the chosen approach, including spectral interference correction, and serve as inherent QC, both for relative instrumental NAA if the Sm/Gd ratio was different for standard and sample as for k_0-NAA. Please also note that given an expanded uncertainty of about 5% on the result there is a good overlap with the BCR-667 certified values.

TABLE II–4. INHERENT QC USING TIME: DETERMINATION OF Gd AND Sm IN A SEDIMENT CRM [II–2]

Sm: Sm-152 -> Sm-153	Mass fraction (mg/kg)				
	Peaks (keV)	01 (1 day)		02 (1 month)	
		Uncorr	Corr	Uncorr	Corr
	103.2	4.96	4.95	55.63	4.95
Weighted Average	4.95		4.95		4.95
Expected StnDev %	0.9%		0.9%		-
Observed StnDev %	-		-		-
Certified Value	4.66 ± 0.20 (k=2)				
Gd: Gd-152 -> Gd-153	Mass fraction (mg/kg)				
	Peaks (keV)	01 (1 day)		02 (1 month)	
		Uncorr	Corr	Uncorr	Corr
	97.4	<306.8	<306.8	4.44	4.44
	103.2	10 274.60	4.20	4.61	4.20
Weighted Average	4.34		-		4.34
Expected StnDev %	14.8%		-		14.8%
Observed StnDev %	2.7%		-		2.7%
Certified Value	4.41 ± 0.12 (k=2)				

II–6. USING THE DIMENSION OF TIME: DETERMINATION OF U IN AN ORE

Four different measurements were carried out for the analysis of U using Np-239: one shortly after irradiation 01, two after a few days (02 and 03) and one after a long decay 04. The first measurement was done at about 25 cm at high dead time of 28% — the second and third were repeats at the same position (but dead time of about 10–17%), while the last measurement was performed at about 5 cm and 1% dead time (see Table II-5). In most cases the NAA operator will choose the second measurement at 277.6 keV for the determination of U due to the half-life of Np-239 of 2.36 d. The fact that all four measurements have an observed weighted standard deviation of 0.8% validates the chosen algorithm for dead time corrections and decay corrections both for relative instrumental NAA and k_0-NAA.

TABLE II–5: INHERENT QC USING TIME: DETERMINATION OF U IN AN ORE [II–2]

U: U-238 -> Np-239		Mass fraction (mg/kg)			
	Peaks (keV)	01 (16 hr)	02 (2 days)	03 (7 days)	04 (3 weeks)
	277.6	4755	4582	4650	4604
Weighted Average	4659				
Expected StnDev %	0.1%				
Observed StnDev %	0.8%				

II–7. USING TIME AND DIFFERENT DECAY SCHEMES: DETERMINATION OF Zn IN AN ORGANIC MATTER SAMPLE

In this experiment four different measurements were carried out for the analysis of Zn: one 16 hours after irradiation, two measurements after a few days and one after a long decay. The result for Zn from the long lived (244.3 d decay) Zn-65 (1739 mg/kg with an observed weighted standard deviation of 0.8%) overlaps with the short lived (decay time 13.76 h) Zn-69m (1755 mg/kg with 2.1% counting statistics overlaps) (see Table II-6). The obtained observed overall StnDev% of 0.7% validates the chosen approach and serves as an inherent QC for correct input data and calculation formulas, potential interferences for both peaks, both for relative instrumental NAA and k_0-NAA.

TABLE II–6. INHERENT QC USING TIME AND DIFFERENT ACTIVATION SCHEMES: DETERMINATION OF Zn [II–2]

Zn: Zn-64 -> Zn-65		Mass fraction (mg/kg)			
	Peaks (keV)	1 (16 hours)	2 (2 days)	3 (7 days)	4 (1 month)
	1115.5	1708	1711	1779	1735
Weighted Average	1739				
Expected StnDev %	0.1%	0.70%	0.2%	0.2%	0.1%
Observed StnDev %	0.8%				

Zn: Zn-68 -> Zn-69m		Mass fraction (mg/kg)			
	Peaks (keV)	1	2	3	4
	438.6	1755	-	-	-
Weighted Average	1755				
Expected StnDev %	2.1%	2.1%			
Observed StnDev %	-				
Overall Zn					
Weighted Average	1740				
Expected StnDev %	0.1%				
Observed StnDev %	0.7%				

II–8. USING FISSION INTERFERENCE CORRECTIONS: DETERMINATION OF La IN AN ORE SAMPLE

In this experiment three different measurements were carried out for the analysis of La: one shortly after irradiation 01, one after a few days 02 and one after a week 03. Normally the NAA operator will choose the second or third measurement mainly taking only into consideration the 1596.2 keV gamma line. Due to fission of U-235 however the results of La will be interfered with. The U fission interference correction at short decays is negligible (0.03 to 0.39 mg/kg), whereas after a long decay it leads to 21.61 mg/kg "apparent" La (see Table II-7).

The fact that after fission interference correction the results of all measurements after different decay times and fission corrections are coherent (notice the very small observed standard deviation of 0.6 %) validates the U result, but also the fission interference correction for La in this case. This is true for k_0-NAA, but also for relative NAA where the standard might have different La/U ratios. Note that if the obtained observed StnDev% would have been large for several elements like La, Zr, Mo and others, this could indicate that the sample does not contain natural U, but that the U is enriched or depleted in the sample.

TABLE II–7. INHERENT QC USING FISSION CORRECTION: DETERMINATION OF La [II–2]

La: La-139 -> La-140	Mass fraction (mg/kg)						
	Peaks (keV)	01 (16 h)		02 (2 days)		03 (1 week)	
		Uncorr	Corr	Uncorr	Corr	Uncorr	Corr
	328.8	158.57	158.54	154.10	153.71	175.65	154.04
	487.0	147.77	147.74	152.59	152.20	173.57	151.96
	815.8	143.22	143.19	151.73	151.34	172.26	150.65
	1596.2	165.83	161.99	152.36	151.97	173.71	152.10
Weighted Average	153.31		155.69		152.14		152.09
Expected StnDev %	0.1%		1.2%		0.1%		0.3%
Observed StnDev %	0.6%		2.8%		0.2%		0.3%

II–9. USING DIFFERENT IRRADIATION CHANNELS AND THRESHOLD INTERFERENCE CORRECTIONS

Table II–8 shows the results for the determination of Mn in an archaeological sample. Three different measurements were carried out for the analysis: 01 after a few hours decay from irradiation channel X, and two measurements shortly after irradiation in a fast rabbit channel 02 and 03. Normally the NAA operator will use the 846.8 keV gamma line for the determination of Mn. However, this value has to be corrected for the $^{56}Fe(n,p)^{56}Mn$ threshold reaction that yields "apparent" concentrations of 61.3 mg/kg and 6.5 mg/kg of Mn, respectively. The fact that now all three results match within counting statistics (the first measurement has lower counting statistics of 5.9% due to the longer decay) both validates the threshold reaction interference correction, the decay corrections, but also the calculated Fe concentration.

TABLE II–8. INHERENT QC USING DIFFERENT TIME AND IRRADIATION CHANNELS: DETERMINATION OF Mn [II–2]

Mn: Mn-55 -> Mn-56	Mass fraction (mg/kg)						
	Peaks (keV)	01 Channel X (2 hours)		02 Channel Y (10 min)		03 Channel Y (30 min)	
		Uncorr	Corr	Uncorr	Corr	Uncorr	Corr
	846.8	221.11	159.79	173.38	166.87	172.20	165.69
	1810.7	222.01	160.69	174.48	167.98	167.08	160.57
	2113.1	222.57	161.24	173.94	167.44	176.01	169.51
Weighted Average	164.11		160.11		167.14		165.09
Expected StnDev %	2.7%		5.9%		0.2%		0.8%
Observed StnDev %	0.3%		0.2%		0.2%		1.1%

REFERENCES

[II–1] DE CORTE, F., SIMONITS, A., Vademecum for k_0-users, DSM Research, Geleen (Netherlands),
www.kayzero.com

[II–2] VERMAERCKE, P., SCK•CEN, Belgian Nuclear Research Centre, private communication (2019).

Annex III

SUMMARY OF POTENTIAL MAJOR ERRORS

This annex contains duplicates of the tables included in the publication, but in a user-friendly format. They are intended to be used in the day to day practice of NAA at research reactors.

TABLE 1. POTENTIAL ERRORS IN SAMPLE HANDLING AND TEST
PORTION PREPARATION AND PROPOSED ACTIONS FOR QUALITY
ASSURANCE AND QUALITY CONTROL

Source of error	Action	
	Quality assurance	Quality control
Interchange of samples	— Use an unambiguous coding system	— Perform a visual check — In case of doubt of sample interchange, check the initial weighted mass of the samples
Insufficient homogenization	— Expand the work instruction for homogenization to include experimental verification through the analysis of replicates — Ask customer for details of sample homogeneity	— Perform a statistical evaluation of the results of five to ten replicates
Moisture content ignored	— Use common sense — Create and use a checklist for review of request — Measure moisture fraction — Check samples for constant weight during drying process	— If drying and moisture correction is applied routinely, check the registration form. Otherwise, none
Element loss during drying	— Ensure appropriate drying temperature for volatile elements — Use common sense — Use freeze drier or desiccant drying technique	None
Hygroscopic behaviour ignored	— Use common sense — Measure moisture fraction	None

TABLE 1. POTENTIAL ERRORS IN SAMPLE HANDLING AND TEST
PORTION PREPARATION AND PROPOSED ACTIONS FOR QUALITY
ASSURANCE AND QUALITY CONTROL (cont.)

Source of error	Action	
	Quality assurance	Quality control
Contamination during production of test portion	— Follow appropriate practices in the laboratory — List sources of contamination and associated elements — Use blanks — Use common sense — Use laminar flow hoods	— Analyse procedural blanks
Weighing; balance malfunctioning (instability, invalid calibration, non-linearity, etc.)	— Perform balance calibration — Conduct regular intermediate performance tests	— Create and use control charts of performance tests and of the periodic (yearly) calibration
Static electricity during weighing	— Use anti-static electricity devices, such as alpha radiation emitting sources	— Perform a visual check
Small sample masses (e.g. a few milligrams) are distributed through the capsule, affecting irradiation and counting geometry	— Use dedicated encapsulation — In some cases glue a few grains to the capsule	— Perform a visual check
Wrong encapsulation material	— Review irradiation requests for elements that may cause problems	None
Sampling handling/test portion preparation	— Perform internal QC: process a portion of a (certified) reference material and a blank capsule simultaneously with the real samples	— Analyse results of control materials and compare with reference values

TABLE 2. POTENTIAL ERRORS DURING CALIBRATION PROCEDURES AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL

Source of error	Action	
	Quality assurance	Quality control
Calibration not acceptable (e.g. to conformity assessment bodies)	— Use, preferentially, CRMs produced in accordance with the requirements of ISO 17034 providing metrological traceability to SI, or otherwise RMs with metrological traceability to an appropriate reference [25, 26][a] — Arrange for calibrations to be performed by conformity assessment bodies	— Check the results of CRM analysis
Invalid calibration status	— Conduct regular performance tests after and between calibrations — Check calibration status on instruments — Add date for recalibration in on-line calendar	— Conduct internal audits — Create and use control charts
Coincidence effects not accounted for in the photopeak efficiency calibration	— Determine the total efficiency curve, enabling coincidence correction calculations — Use validated software [26][b]	— Check the results of RM analysis and proficiency testing — Use inherent QC of NAA (see Annex II) — Compare the ratio of coincidence-free radionuclides (e.g. Cr-51, Zn-65) with non-coincidence-free radionuclides (e.g. Co-60, Se-75) at different positions from the detector

TABLE 2. POTENTIAL ERRORS DURING CALIBRATION PROCEDURES AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL (cont.)

Source of error	Action	
	Quality assurance	Quality control
Voluminous efficiency not accounted for in the photopeak efficiency calibration	— Use validated software or appropriate Monte Carlo software packages	— Check the results of CRM analysis and proficiency testing
Inadequate fit of the photopeak efficiency	— Use validated software	— Verify the chi-square of the residuals of the fit — For k_0-NAA: analyse appropriate (synthetic) multi-element materials [27]
Unacceptable certificates of calibration sources	— Describe criteria for acceptable certificates — Conduct incoming check of all calibration certificates	— Conduct internal audits

[a]   ISO 17034 [25] specifies general requirements for the competence and consistent operation of RM producers. It covers the production of all RMs, including CRMs, and aims at confirming or recognizing the competence of RM producers. According to ISO/IEC 17025 [26], in order to ensure that measurement results are traceable to the International System of Units (SI), values of CRMs provided by a competent producer can be used. RM producers fulfilling the requirements of ISO 17034 are considered to be competent.

[b]   ISO/IEC 17025, paragraph 7.11.2 [26], adds in Note 2 that "Commercial off-the-shelf software in general use within its designed application range can be considered to be sufficiently validated." However, it is also recommended in paragraph 7.11.2 that "Whenever there are any changes, including laboratory software configuration or modifications to commercial off-the-shelf software, they shall be authorized, documented and validated before implementation." The latter may also be derived from its fitness for purpose demonstrated in scientific publications.

TABLE 3. POTENTIAL ERRORS IN SAMPLE HANDLING AND TEST PORTION PREPARATION FOR CALIBRATION PURPOSES AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL

(in addition to those in Table 1)

Source of error	Action	
	Quality assurance	Quality control
Differences in the isotopic abundance of some elements in real samples and some elements in standards	— Refer to checklist upon receipt of samples that contain elements that may have isotopic abundance different from the standards — Use RMs from producers conforming to ISO 17034	— None, except for U-235 through measuring the ratio of fission product activity and Np-239 activity — Use inherent QC of NAA (see Annex II)
Stoichiometry of pure element standards differs from expectation	— Use CRMs of single element standards	None
Mass of the (certified) RM used is too small	— Use minimum mass as prescribed in the RM's certificate	— Conduct independent verification of masses used
Differences in filling the height of material in capsules	— Measure filling heights of samples and standards — Pelletize samples and calibrators	— Verify filling or pellet height — Density (for solid materials): verify with the material composition and published data on density
Certificates of calibrators do not meet metrological requirements	— Define criteria in the work instruction — Verify the certificates of the calibrators (e.g. on the web sites of producers)	— Inspect existing certificates
Interference by impurities in self-made calibrators	— Verify the purity of the substance before use	— Check analysis results

TABLE 4. POTENTIAL ERRORS IN NEUTRON FLUX MONITOR
PREPARATION AND PROPOSED ACTIONS FOR QUALITY
ASSURANCE AND QUALITY CONTROL
(in addition to those in Tables 1 and 2)

Source of error	Action	
	Quality assurance	Quality control
Weighing of flux monitors	— Perform balance calibration — Conduct regular intermediate performance tests	— Create and use control charts of performance tests
Mass (fraction) of comparator/monitor	— Buy from RM producers conforming to ISO 17034	— Create and use control chart of flux at fixed positions and thermal–epithermal ratio
Neutron flux gradient in irradiation container underestimated	— Sandwich sample test portions between neutron fluence rate/calibrator test portions	— Check analysis results of CRMs — Check for abnormal results in the flux results (see Section 6.2)
Neutron spectrum gradients in irradiation container underestimated	— Use monitors sensitive to detecting differences between epithermal and fast neutron fluence rates	— Check analysis results of CRMs for elements with a high resonance integral
Contribution of fast neutrons underestimated	— Select and add an appropriate flux monitor for fast neutrons	— Check analysis results of CRMs for elements with activation products through fast neutron reactions

TABLE 4. POTENTIAL ERRORS IN NEUTRON FLUX MONITOR
PREPARATION AND PROPOSED ACTIONS FOR QUALITY
ASSURANCE AND QUALITY CONTROL
(in addition to those in Tables 1 and 2) (cont.)

Source of error	Action	
	Quality assurance	Quality control
Non-repeatable or non-reproducible results for thermal–epithermal flux ratio and alpha parameter	— Select and use an appropriate flux monitor depending on the degree of neutron thermalization in the facility	— Check analysis results of CRMs for elements with a high resonance integral — Create and use control chart for thermal–epithermal flux ratio and alpha parameter — Analyse appropriate (synthetic) multielement materials [27]

TABLE 5. POTENTIAL ERRORS DURING THE IRRADIATION
PROCEDURE AND PROPOSED ACTIONS FOR QUALITY ASSURANCE
AND QUALITY CONTROL

Source of error	Action	
	Quality assurance	Quality control
Thermal/epithermal/fast neutron flux gradients	— Sandwich samples and flux monitors	— Check results of flux monitors to observe flux gradients
Strong neutron absorbers	— Anticipate during review of request for analysis	— Observe possible neutron flux depression and lower flux in the flux monitors surrounding the sample — Observe possible deformation or melting of plastic capsules due to heating that may occur in the presence of exceptionally high amounts of boron
Imprecise timing of duration of irradiation	— Mostly for NAA with short half-life radionuclides: Verify imprecision experimentally — Other types of NAA: Use internal QC samples and calibrator/ fluence rate monitors	— Short half-life NAA: Observe systematic differences in the trueness of the results obtained for short half-life radionuclides — Long half-life NAA: Compare results of different radionuclides from the same element
Element losses due to volatilization and migration through plastics (e.g. mercury, bromine)	— Use common sense — Encapsulate in quartz — Include an ionic liquid such as thiocyanate on a filter paper within the same encapsulation	— Use blanks

TABLE 5. POTENTIAL ERRORS DURING THE IRRADIATION
PROCEDURE AND PROPOSED ACTIONS FOR QUALITY ASSURANCE
AND QUALITY CONTROL (cont.)

Source of error	Action	
	Quality assurance	Quality control
Neutron self-attenuation (thermal, epithermal), neutron flux depression	— Use common sense — Request review checklist — Use validated software such as MATSSF [31]	— Sandwich samples between flux monitors to provide information if the unperturbed flux gradients are known
Recoil contamination of samples	— Perform advance analysis of encapsulation material	None
Burnup of target isotope	— Use common sense — Set maximum irradiation time	— Analyse results of internal QC samples

TABLE 6. POTENTIAL ERRORS DURING THE DECAY PERIOD AND
PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY
CONTROL

Source of error	Action	
	Quality assurance	Quality control
Incorrect synchronization of clocks at the reactor and in the counting room	— Use radiofrequency controlled clocks — Verify synchronization regularly	— Use analysis results of RMs and correlation of deviations with half-lives of the radionuclides measured
Daylight saving time	— Irradiate and count in the same period — Use common sense	— Use analysis results of RMs and correlation of deviations with half-lives of the radionuclides measured
Wrong correction of decay before and during measurements	— Use validated software	— Use inherent QC of NAA (see Annex II)

TABLE 7. POTENTIAL ERRORS IN THE MEASUREMENT OF THE
GAMMA RAY SPECTRUM AND PROPOSED ACTIONS FOR QUALITY
ASSURANCE AND QUALITY CONTROL: SPECTRUM

Source of error	Action	
	Quality assurance	Quality control
Peak shifts and doublets in the spectrum owing to gain instability	— Use spectrometer performance control and control charts for peak position at low, medium and high energy — Ensure proper management of spectrometers[a]	— Check for the existence of peaks in the spectrum that do not match energy calibration
Increased spectral interferences owing to poor detector resolution	— Use spectrometer performance control and control charts for FWHM at low, medium and high energy. If needed, also for FWTM — Ensure proper management of spectrometers[a]	— Perform a visual check of the results of spectrum fitting
Non-Gaussian shaped peaks	— Use spectrometer performance control and control charts — Ensure proper management of spectrometers[a]	— Perform a visual check of the results of spectrum fitting (peak tailings may result in poor fits or satellite peaks) — Look at residuals after peak fitting
Gamma ray self-attenuation	— Use common sense — Make first order estimates on basis of estimate of major element composition	— Check that the results of RM analysis and inspection if results measured on low energy gamma rays (e.g. <200 keV) concur with results measured for the same radionuclide at higher energy gamma rays — Prepare and use appropriate (synthetic) multielement materials [27]

[a] See also Ref. [39] and section I, Instrumentation, of the NAA e-learning course [19].

TABLE 8. POTENTIAL ERRORS IN THE MEASUREMENT OF THE GAMMA RAY SPECTRUM AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL: DETECTION

Source of error	Action	
	Quality assurance	Quality control
Wrong source–detector distance	— Minimize the possibility of measuring at different distances (e.g. by using fixed measurement positions)	— Check dead time (too high if counting too close) — Check whether there are peak shifts in the measured spectrum or whether peaks are too small owing to high count rates
Wrong efficiency curve selected	— Use automatic read-out of the measuring position	— Use results of RM analysis — Use appropriate (synthetic) multielement materials [27]
Geometry (size) differences between calibration, sample and flux monitor	— Fill height registration and correction — Immobilize test portions inside their encapsulation — Use validated software	— Perform a visual check
Dead time variations during counting	— Zero dead time system/ loss free counting — Set a maximum dead time based on a double source validation technique	— Perform a visual check during measurement
Coincidence summing	— Calculations, measurement at larger source–detector distance — Measure sample and calibrator at same distances and count rates — Use validated software	— Use results of RM analysis and inspection for consistent results on all gamma rays emitted by the same radionuclides, especially for radionuclides decaying by gamma rays in cascade (e.g. Co-60, Se-75)

TABLE 8. POTENTIAL ERRORS IN THE MEASUREMENT OF THE
GAMMA RAY SPECTRUM AND PROPOSED ACTIONS FOR QUALITY
ASSURANCE AND QUALITY CONTROL: DETECTION (cont.)

Source of error	Action	
	Quality assurance	Quality control
Contamination of detector end cap	— Cover detector end cap with household wrapping foil — Always keep spacer between source and end cap	— Measure detector background with no sources present — Create and use control chart of background for well chosen regions of interest

TABLE 9. POTENTIAL ERRORS IN GAMMA RAY SPECTRUM INTERPRETATION, VALIDITY CHECKING AND REPORTING AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL

Source of error	Action	
	Quality assurance	Quality control
Calculation errors	— Use validated calculation sheets or commercial software codes (validated in international literature)	— Use the results of CRM analysis and proficiency testing — Use inherent QC of NAA (see Annex II)
Peaks not assigned to radionuclides	— Use spectrometer performance control to avoid drifts — Verify decay time independently	— Use the results of RM analysis and proficiency testing — Use inherent QC of NAA (see Annex II)
Intensity ratios between peaks do not match theoretical values	— Prevent counting close to end cap (and coincidence effects) — Include verification of efficiency curve in method validation	— Use the results of RM analysis and proficiency testing — Use inherent QC of NAA (see Annex II)
Interfering nuclear reactions	— Use common sense — Calibrate	— Use the results of RM analysis and proficiency testing — Use inherent QC of NAA (see Annex II)
Typos and transposing errors; interchange of samples and customer codes	— Verify all manually entered data independently	— Laboratory management performs final verification and authorization — Use inherent QC of NAA (see Annex II)
Contamination	— Use a blank	— Create a control chart of the blank measurements

TABLE 9. POTENTIAL ERRORS IN GAMMA RAY SPECTRUM
INTERPRETATION, VALIDITY CHECKING AND REPORTING AND
PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY
CONTROL (cont.)

Source of error	Action	
	Quality assurance	Quality control
Neutron flux gradient correction	— Prescribe sandwiching of samples and flux monitors	None
Trueness unclear	— Use CRM with each batch, even if the batch consists of one sample	— Use the results of CRM analysis and proficiency testing
Unsatisfactory results	— Perform non-conformance reporting and root cause analysis	— Participate in proficiency testing
Reporting format differs from customer request	— Ensure effective internal communication	— Evaluate customer satisfaction
Reporting beyond deadline	— Incorporate a safety margin in planning	— Conduct internal audits — Establish a key performance indicator on reporting time
Reporting results even though results of control materials are not satisfactory	— Laboratory management performs final verification and authorization	— Conduct internal audits

TABLE 10. EXAMPLE OF A CHECKLIST FOR THE ASSESSMENT
OF THE FEASIBILITY OF AN ANALYSIS REQUEST FOR NEUTRON
ACTIVATION ANALYSIS

Questions to customer	Issues to consider
Specific elements or panoramic analysis	
Indication of the expected mass fractions	
Any indication of interfering elements (which interfering elements varies on a case-by-case basis)	
Requested degree of trueness and uncertainty Requested limit of detection	— Performance indicators for the methods used at the laboratory (e.g. specificity and selectivity, trueness, precision, robustness, uncertainty, traceability) — Contamination risks during sample preparation and irradiation
Date of reporting	— Availability of reactor — Availability of human resources for all parts of the procedure — Availability of equipment within the time window needed for performing the analysis
Available amount of sample	— Preferably at least twice the amount the laboratory normally uses for analysis
Any indication of the degree of homogeneity	
Possible moisture fraction	
Presence of components (e.g. proteins) that may decompose during irradiation, causing pressure buildup Presence of significant amounts of neutron absorbing elements (e.g. boron, lithium, samarium, gadolinium, iridium) Presence of significant amounts of gamma ray absorbing elements (lead, thallium, bismuth) Presence of uranium (fission)	— If the customer is not aware of such components and/or elements, and the presence of significant amounts cannot be excluded, the laboratory may have to perform a series of tests (such as gamma ray transmission), or test irradiations with increasing amounts of material; to consider analysis by an alternative method (such as X ray fluorescence analysis); or to consider rejecting the request for analysis

TABLE 10. EXAMPLE OF A CHECKLIST FOR THE ASSESSMENT
OF THE FEASIBILITY OF AN ANALYSIS REQUEST FOR NEUTRON
ACTIVATION ANALYSIS (cont.)

Questions to customer	Issues to consider
Aspects of safety in material handling (e.g. carcinogenic, contagious), safeguards	
Special reporting format requirements	
Accept price and delivery conditions (if any)	

TABLE 11. POTENTIAL ERRORS IN THE ANALYTICAL PROTOCOL AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL

Source of error	Action	
	Quality assurance	Quality control
Wrong combination of sample size, irradiation, decay, measurement times and counting geometry	— Maintain a database of past measurements — Study literature — Test irradiations — Use advanced computer prediction programs	None

TABLE 12. POTENTIAL ERRORS IN THE MANAGEMENT OF THE NEUTRON ACTIVATION ANALYSIS LABORATORY AND PROPOSED ACTIONS FOR QUALITY ASSURANCE AND QUALITY CONTROL

Source of error	Action	
	Quality assurance	Quality control
Details of how past measurements were performed are not known	— Register and record all conditions/parameters	— Conduct internal audits
Confusion on how work has to be done and what has to be registered	— Refer to flow charts, forms and training material	— Conduct internal audits
Recurring errors and mistakes	— Register and manage non-conformity — Maintain and check an experience database	— Conduct internal audits
Random decisions at go/no-go points	— Define objective assessment criteria	— Conduct internal audits

TABLE 13. POTENTIAL ERRORS DURING PARTICIPATION IN
PROFICIENCY TESTING EXERCISES IN THE NEUTRON ACTIVATION
ANALYSIS LABORATORY AND PROPOSED ACTIONS FOR QUALITY
ASSURANCE AND QUALITY CONTROL

Source of error	Action	
	Quality assurance	Quality control
Non-conformity owing to wrong report format	— Conduct independent verification before submitting results	— Laboratory management performs final verification
Results not accepted because report received after deadline	— Incorporate a safety margin into planning	— Conduct internal audits
No conclusions possible after receipt of report from the proficiency testing provider because of too few results and/or absence of information on techniques used	— Prepare work instructions for selection of proficiency testing schemes	None
Acceptance of provider's report plus follow-up on non-conformity without any self-assessment by the laboratory	— Prepare work instruction for self-assessment (e.g. based on ζ score instead of z-score)	— Conduct internal audits
Only NAA laboratory; all others use 'destructive' techniques	— Prepare work instructions for selection of proficiency testing schemes	None