

DIPLOMARBEIT

Evaluierung eines Neutronenaktivierungsanalysators

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unter Anleitung von

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durch

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Contents

1	Preface	4
2	Theory	6
2.1	Neutron Capture	6
2.2	Detectors and Instrumentation	8
2.3	Loss Free Counting	16
2.4	Activation Analysis	18
2.4.1	Error calculation	19
2.5	Separation of Lines of Similar Energies	20
3	User's Manual	22
3.1	Step-by-step Guide	22
3.2	Components	23
3.2.1	Pneumatics	24
3.2.2	Sample Changer	25
3.2.3	Electronics	26
3.3	Software	28
3.3.1	GSPEC	29
3.3.2	NDANALYZ	34
3.3.3	NDANAEX	37
3.3.4	Other Utilities	37
3.4	Measurements	39
3.4.1	Manual	39
3.4.2	Semi-automatic	39
3.4.3	Fully Automatic	41
3.4.4	Further Possibilities	43
3.5	Library	44
3.6	Interpretation/Caveats	45
3.6.1	Input Values	45
3.6.2	Output Values	46

4	Evaluation	47
4.1	System performance	47
4.1.1	Mechanics	47
4.1.2	Detection System	49
4.1.3	Reactor	52
4.1.4	Software	53
4.2	Sample data	54
5	Conclusion	56

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Last but not least, my wife, Elisabeth.

Chapter 1

Preface

In April 2003 I started working on the Neutron Activation Analyzer during my project work. I helped to develop the sample changer and parts of the circuitry and spent a lot of time trying to understand and debug the software.

I also did a lot of measurements, to understand how the system works as well as for performance testing.

Working consistently on a single measurement system for over a year brings intimate knowledge of its behavior, the inner workings and its limits. I had to learn how to use all of the systems capabilities to their fullest extent with a lot of experimenting.

A flexible system with a wide range of options naturally tends to be complex and not easy to use. I spent a lot of thought and effort on the task of making the usage of the system as simple as possible, as the intention of an automated system has to be. I had several opportunities to explain the automated processes to fellow students and professors, both in English and German. While my understanding of the Neutron Activation Analyzer grew, explaining became harder because I tried to cover every possible option the system offers.

This diploma work is intended to cover two major tasks:

- to offer a user's manual for everyone who intends to use the Neutron Activation Analyzer, accurately explaining every possible task the system can perform.
- to evaluate the Neutron Activation Analyzer and show its capabilities and limits.

A users manual has to offer an easy starting point for the inexperienced user while at the same time explain all the possible options for the experienced experimenter. I tried to bridge this large gap in chapter 3.

Chapter 4 gives the evaluation of the Neutron Activation Analyzer using data collected during my time at the system.

Chapter 2

Theory

2.1 Neutron Capture

When a sample is brought in the core of a reactor, it will be irradiated by a constant neutron flux. Depending on the contents of the sample, several nuclear reactions occur. If, for example, a sample of 1 g NaCl is irradiated and we are interested in the possible reactions, we have to find out, which nuclides are present.

NaCl consist of Na and Cl, where stable Na only occurs in nature as ^{23}Na and Cl consist of 75,77%, [6] ^{35}Cl and of 24,23% ^{37}Cl . So, with the molar weight of Na being 22,989768 g and the molar weight of Cl being 35,4527 g, 1 g of NaCl consists of 0,3934 g ^{23}Na , 0,1470 g ^{37}Cl , and 0,4596 g ^{35}Cl as can be seen in table 2.1.

With thermal neutrons, the main nuclear reaction for ^{23}Na and ^{37}Cl is the (n, γ)-reaction. Here, a neutron is captured within the nucleus of the nuclide and spontaneous γ -radiation is emitted. The nuclide retains the neutron and becomes a different, unstable nuclide. The same reaction is possible for ^{35}Cl , but two other reactions can occur: an (n, α)- and an (n, p)-reaction. In each case a neutron is captured but instead of the γ -radiation a α -particle or a proton is emitted, respectively.

All these nuclear reactions lead to an unstable nuclide which decays to

nuclide	mass [g]
^{23}Na	0,3934
^{35}Cl	0,4596
^{37}Cl	0,1470

Table 2.1: Nuclides contained in 1 g of NaCl

original nuclide	nuclear reaction	unstable nuclide	decay	stable nuclide	cross-section σ [barn]
^{23}Na	(n, γ)	^{24}Na	β^-	^{24}Mg	0,43
^{35}Cl	(n, p)	^{35}S	β^-	^{35}Cl	0,4
^{35}Cl	(n, α)	^{32}P	β^-	^{32}S	0,00008
^{35}Cl	(n, γ)	^{36}Cl	β^-	^{36}Ar	43,7
^{35}Cl	(n, γ)	^{36}Cl	β^+	^{36}S	43,7
^{37}Cl	(n, γ)	^{38}Cl	β^-	^{38}Ar	0,42

Table 2.2: Possible nuclear reactions for NaCl with their respective cross-sections [6]



Figure 2.1: Reaction chain for ^{23}Na and ^{37}Cl

a stable nuclide. The whole reaction can be found in table 2.2. Figures 2.1 and 2.2 show the nuclear reactions and their end products.

We are also interested in the final activity of the sample after radiation. For safety reasons it is of great importance to have at least a good estimate of the possible activity of a sample.

The activity of a sample is the sum of the activities of all the unstable nuclides in the sample. With N_0 the number of atoms of the original nuclide, its cross-section σ and neutron flux ϕ and the number of atoms of the produced unstable nuclide $N(t)$ with the decay constant λ we can write:

$$\frac{dN(t)}{dt} = N_0\phi\sigma - \lambda N(t) \quad (2.1)$$

Solving this differential equation we arrive at

$$\lambda N(t) = N_0\phi\sigma (1 - e^{-\lambda t}) = A(t) \quad (2.2)$$

with the activity A .

Collecting $N_0\phi\sigma$ into A_s we call A_s the saturation activity because the

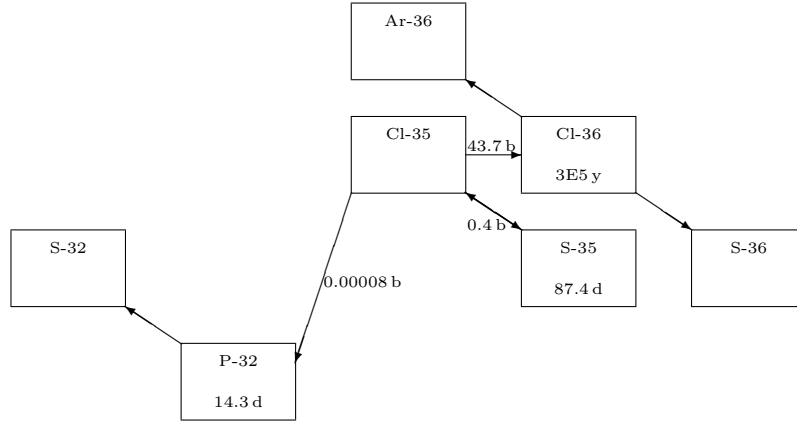


Figure 2.2: Reaction chain for ^{35}Cl

nuclide	τ	$A[s^{-1}]$
^{24}Na	15 h	$1,3 \cdot 10^6$
^{36}Cl	301000 a	4,14
^{38}Cl	37,21 min	$56,6 \cdot 10^6$
^{35}S	87,5 d	52056
^{32}P	14,26 d	64

Table 2.3: Expected activities of the nuclides of 1 g NaCl after irradiation for 10 s at a flux of $3 \cdot 10^{12} \text{ cm}^{-2}\text{s}^{-1}$

activity of the nuclide will eventually reach A_s , as can be seen in figure 2.3. We can see that the activity is related to the nuclide's decay constant λ and therefore to its half life τ with $\lambda = \frac{\ln 2}{\tau}$. A nuclide with a very long half life (i.e. ^{36}Cl with $\tau = 3 \cdot 10^5 \text{ a}$) can only be produced in significant amounts by irradiating its mother nuclide (^{35}Cl) for very long times. Another important factor is the cross section for the nuclear reaction.

Using those initial considerations we can see that the activity of 1 g of NaCl after irradiation of 10 s will be dominated by ^{38}Cl and ^{24}Na . The next candidate will be ^{35}S . Considering a neutron flux of $\phi = 3 \cdot 10^{12} \text{ cm}^{-2}\text{s}^{-1}$ we can calculate the activities which can be found in table 2.3.

2.2 Detectors and Instrumentation

Figure 2.4 shows the internals of a modern detection system. Information collected in the detector is amplified in the pre-amplifier and amplifier and filtered before being collected and recorded in a multichannel analyzer.

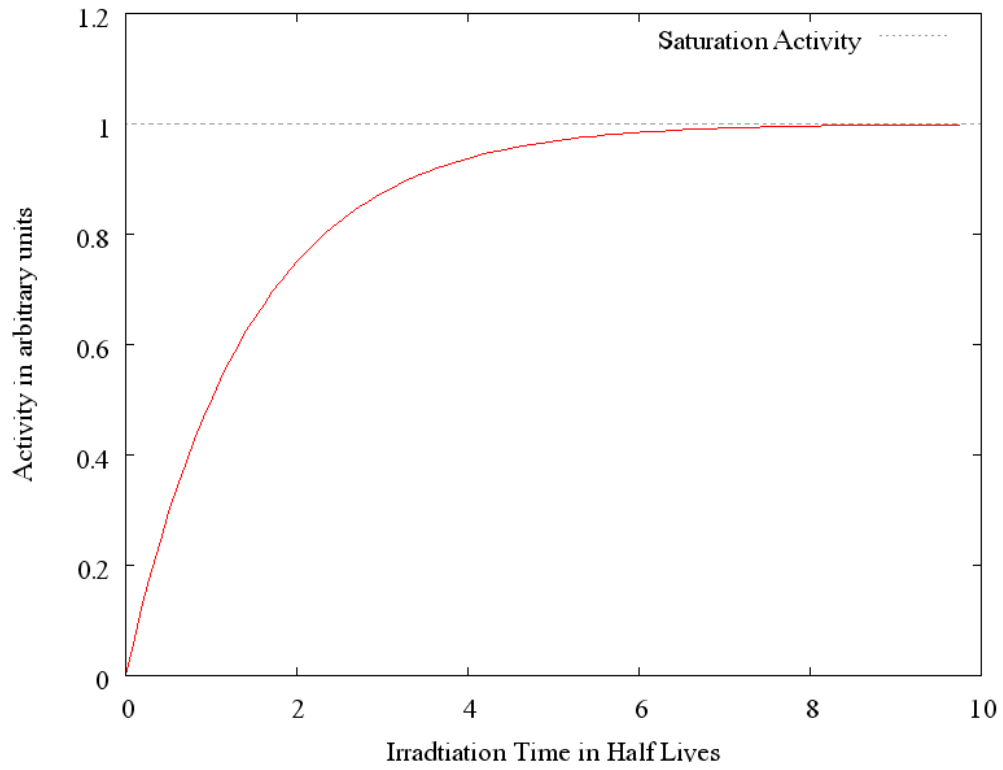


Figure 2.3: Activity after irradiation. The activity eventually reaches the saturation activity



Figure 2.4: Detector chain, following the information from detection to recording and storage in the Multi Channel Analyzer

To detect electromagnetic radiation, its interaction with a medium is measured. Several interactions with matter are possible, the easiest to detect and measure is ionization, as here charge carriers are produced which can be measured easily and quantitatively. To measure the energy of some radiation, it is necessary for this radiation to be completely absorbed within the detector medium. Therefore, a comparatively dense medium is necessary for highly penetrating radiation like γ - and X-rays.

Instead of using gas as a medium like in the ionization chamber, the proportional counter or the Geiger-Müller counter, either ^{14}Si or ^{32}Ge with high density and moderate to high atomic numbers are used in γ -detection.

Both fulfill the following prerequisites for quantitative measurements of radiation:

- a minimum of free charge carriers not generated by radiation
- no charge trapping mechanisms to guarantee longer carrier life time than charge carrier collection time
- total absorption of radiation energy within the active volume of the detector.
- the energy for generation of one charge carrier should be constant and independent of energy or type of radiation

During ionization, the number of ion pairs n produced by an amount of absorbed energy E follows equation 2.3, where w is the mean energy required for the production of one ion pair and $F < 1$ is the *Fano-factor*.

$$n = \frac{E}{w} \pm \sqrt{Fn} \quad (2.3)$$

As mentioned above, the constancy of w is a necessary prerequisite for radiation energy detection, energy resolution is determined primarily by the statistical fluctuation of n as described by the standard deviation \sqrt{Fn} .

The Fano-factor can be related to the inverse of the ionization probability $\eta = E_i/w$, where E_i is the ionization potential or energy gap, describing the efficiency of the ionization process as well as the influence of competing mechanisms as excitation or phonon production.

For a vanishing ionization probability, F will reach its upper limit of 1 indicating Poisson statistics and thus statistical independence of individual ionization events. For increasing η though, the correlation of ionization events increases accordingly, reaching an exact apportioning of radiation energy to the production of charge carrier pairs with vanishing statistical fluctuation.

While in gas-filled detectors the ionization energy lies around 35 eV, only 2.8 eV in Ge and 3.6 eV in Si are necessary to move an electron to the conduction band, creating a electron-hole pair of high and almost equal mobility. Thus, a higher number of charge carriers for a given energy is produced, resulting in reduced statistical fluctuation and a larger electric signal.

As direct recombination of charge carriers is very infrequent and mostly due to trapping at crystal impurities in semiconductors, sufficient life time is provided in state-of-the-art high purity detectors.

Free charge carrier concentration is too high for detector applications by several orders of magnitude, as it is governed by the net concentration of donor and acceptor impurities. The only known semiconductor device where conductivity is exclusively determined by the intrinsic mechanism of the ideal semiconductor is the reverse biased PN or *Schottky* junction. Here, because of the applied bias voltage, all free charge carriers are removed from the *depletion layer* and new charge carriers generated within this zone are collected in the outer circuit as signal or leakage current. Therefore the width of the depletion layer is the active zone of a semiconductor detector and is proportional to $\sqrt{V/N}$, where V is the applied reverse bias and N the net donor/acceptor impurity concentration.

For γ - or X-ray detection, extended depletion layers are required, providing detector volumes of up to 200 cm³. Larger depletion layers may only be obtained by a further reduction of net impurity concentrations, as bias voltage is limited by electrical breakdown. Old detectors could only provide sufficiently low net impurity concentrations by compensating residual impurities with impurities of the opposite type. Using the *lithium drift process*, a PN junction is formed by lithium diffusion and back-biased at room temperature where lithium has very high mobility. Lithium drifts in a way that, under the influence of internal fields, a uniform electric field is produced. In the central region, this means that there is no net space charge left, i.e. the number of lithium ions compensates the number of p-type impurities exactly. With the lithium drift process, depletion depth is only limited by drift depth, which is 0.5 cm in silicon and 2 cm in germanium. To prevent redistribution of the drift profile, lithium drifted detectors must be stored at low temperatures.

Since the advent of high-purity germanium with impurity concentration less than $1 \cdot 10^{-12}$, lithium drifting is no longer necessary, giving the most obvious advantage of easier storage of the detectors. Another important advantage is the absence of inactive detector volume, making the detector more suitable for high counting rates.

All information from radiation detectors is carried by minute packets of

charge (10^{-16} to 10^{-12} C) presented as short (10^{-9} to 10^{-6} s) pulses of current randomly occurring in time. To extract this information, precise charge measurements of individual events with optimum signal-to-noise ratio have to be performed as fast as possible in order not to interfere with preceding or subsequent events. The result has to be presented as a digital quantity to enable counting, sorting and permanent recording.

Absorbed energy in the active detector volume creates charge carriers with a total charge of exactly the same energy. Those are collected under the influence of an electric field as a short pulse of current $i(t)$, where the pulses area $q = \int i(t) dt$ carries the spectroscopic information on the irradiation quantum. Thus, during amplification, this information must never change.

The pulse response $H(t)$ of the amplification system, i.e. the preamplifier and a subsequent main amplifier, describes the shape of the output pulse in response to the delta function. The output pulse $O(t)$ in response to a real input pulse is obtained by convolution of $i(t)$ with $H(t)$. During convolution, pulse area is conserved.

Noise energy is proportional to system bandwidth, so to minimize the influence of the electronic noise of the amplifier, controlled additional bandwidth limitation is necessary. Therefore in practical systems $H(t)$ is a low-pass transfer function characterized by a corner frequency ω_C and a shaping time constant $\tau = \frac{1}{\omega_C}$.

The pulse response of a low-pass filter with infinite number of elements to a delta function is a gaussian shape. For finite numbers of elements, the gaussian shape becomes the less symmetrical semi-gaussian type (see figure 2.5).

Besides the electronic noise of the amplifier, there are also sources for so called ‘parallel’ noise, namely shot noise from the statistical fluctuation of detector and amplifier leakage currents and thermal noise from resistive components connected to the input of the preamplifier. These noise currents cannot be distinguished from signal current and must be minimized by suitable design measures. Parallel noise current contribution increases with the duration of $H(t)$ and the signal-to-parallel-noise ratio is worsened by bandwidth reduction. Therefore, for a given detector-amplifier combination, there exists an optimum shaping time constant which results in minimum total noise.

For pre-amplification, a gated transistor reset amplifier is used, as can be seen in figure 2.6. The preamplifier changes the pulses $i(t)$ coming from the detector to a step-function, where each step resembles the charge of the collected pulse q . When the upper limit of the linear operating range is reached, the switch discharges the capacitor, creating a step-function similar to figure 2.7

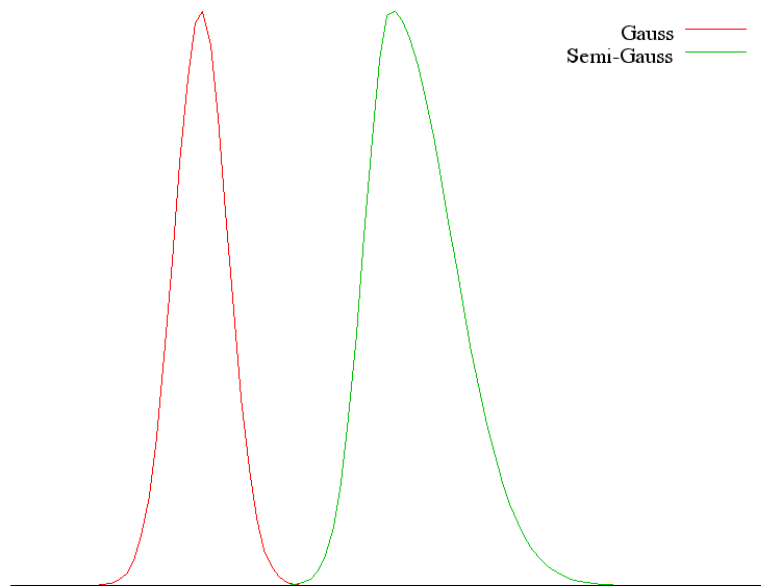


Figure 2.5: Gaussian and Semi-Gaussian shape

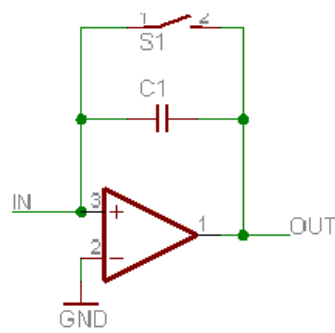


Figure 2.6: Diagram of a gated transistor reset preamplifier

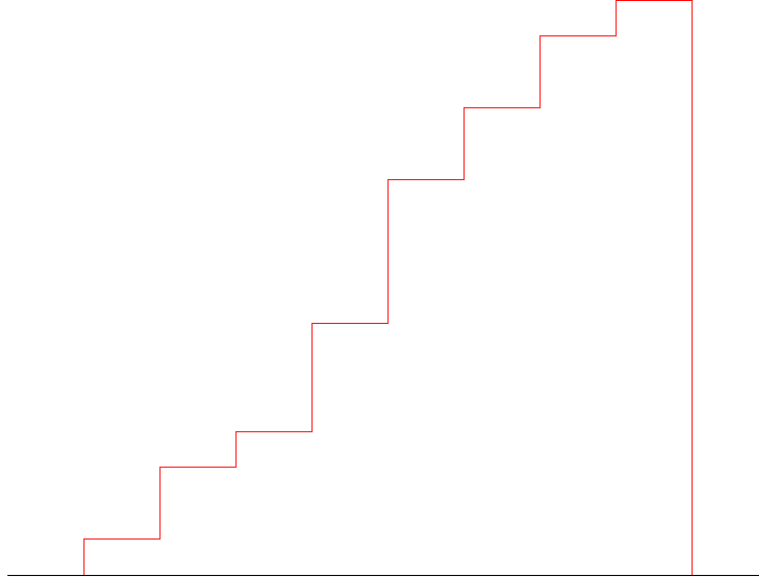


Figure 2.7: Output signal of the gated transistor reset amplifier.

Instead of a main amplifier and subsequent filter, the current system of the Neutron Activation Analyzer uses the so called *Preloaded Digital Filter* [14], developed at the Atominstut in Vienna is used. A block diagram of the PLDF can be seen in figure 2.8. The filter performs pulse shortening and pole zero cancellation in the analog domain, making the use of cheap monolithic ADCs possible.

The pseudorectangular prefilter [3, 12] with a pulse duration of $1.5 \mu\text{s}$ has been chosen for further optimization of signal to noise ratio (see figure 2.9).

The Preloaded Digital Filter also performs calculation of weighting factors for Loss Free Counting [14]. The filter acquisition time AT is system dead-time and pileup sensitive period at the same time. The probability of dead-time escape PDE and of pulse pileup escape PPE therefore are equal and become:

$$PDE = PPE = e^{-AT IR} \quad (2.4)$$

with IR the true input counting rate.

Dead-time correction can thus be performed by using the inverse of PDE as weighting factors to be added to the channel addressed during the measurement. By extending AT to $2AT$ by means of a monostable multivibrator, the pulse processing probability $PPP = PDE \cdot PPE$ can be calculated. Its inverse produces weighting factors for complete loss correction.

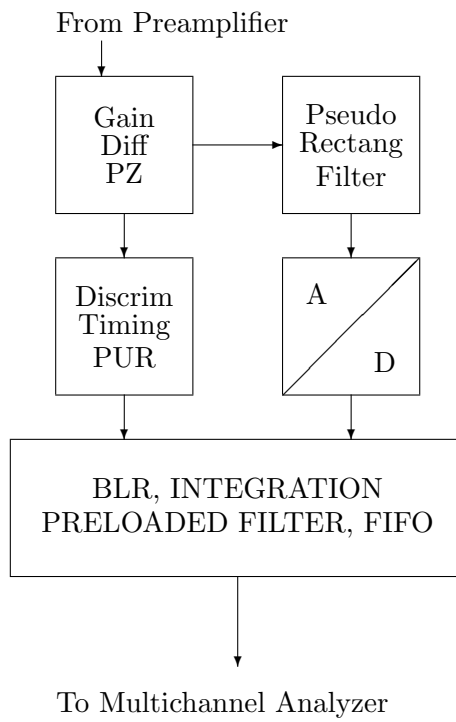


Figure 2.8: Block diagram of PLDF with pulse shortening and pole zero cancellation in the analog domain

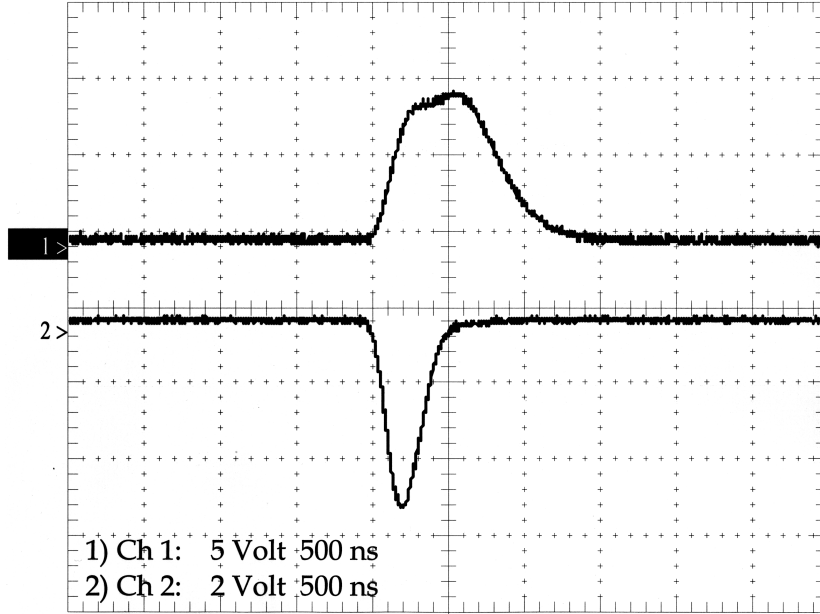


Figure 2.9: Pulse response of pseudorectangular filter (1) and preceding differentiator (2)

2.3 Loss Free Counting

Loss Free Counting was first introduced by Westphal [8, 9]. With the introduction of the *Virtual Pulse Generator Method* [10, 11], the first quantitative correction of dead-time and pileup losses was accessible.

The *Virtual Pulse Generator Method* was based on the extension of *System Busy* by means of a monostable multivibrator and soon became the state-of-the-art solution for all kinds of nuclear pulse spectroscopy at high and variable counting rates.

Complete loss correction (*CLC*) is the product of dead-time correction (*DTC*) and pileup correction (*PUC*). Both, dead-time correction and pileup correction are themselves functions of the dead-time d and pileup time p , respectively, and can be written as

$$DTC = e^{ad} \quad PUC = e^{ap} \quad (2.5)$$

with the input counting rate a .

The extension of dead-time to correct for pileup losses can easily be justified by collecting the two exponents into *CLC*:

$$CLC = DTC \cdot PUC = e^{ad} e^{ap} = e^{a(d+p)} \quad (2.6)$$

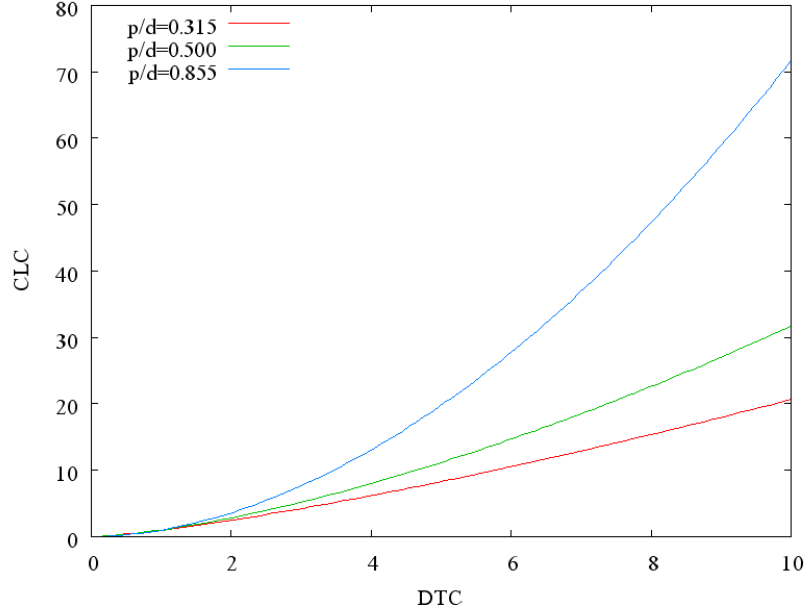


Figure 2.10: Lookup tables showing DTC versus CLC for different values of p/d . See equation 2.7

The extension of dead-time may be performed by manually adjusting monostable multivibrators [10, 5] or by computer controlled digital delay generators.

In case of the Neutron Activation Analyzer however, dead-time correction factors are produced by the *Preloaded Digital Filter* without extended AT and pileup correction is computed by the software implementation of a multichannel analyzer, GSPEC [16, 17, 15, 13].

Rewriting equation 2.6 in terms of DTC (see equation 2.7), it is easy to see how GSPEC, using DTC arriving from the filter and the constant ratio of pileup sensitive time p and dead-time d calculates CLC .

$$CLC = DTC^{(1+\frac{p}{d})} \quad (2.7)$$

To enable real-time calculation of correction factors, a lookup table is used instead of performing the above calculation. Figure 2.10 shows lookup tables for different values of p/d .

The ratio of pileup to dead-time is adjustable by means of the setup-screen in GSPEC (see section 3.3.1, figure 3.7).

2.4 Activation Analysis

To analyze the contents of a given sample the sample is irradiated with neutrons. The nuclear reactions (see section 2.1) activate the stable nuclides. These activated nuclides can be measured with a detector and can be related to the stable mother nuclides.

After irradiation of a sample the activity of any given nuclide with the decay constant λ contained within the sample can be written as (see equation 2.2):

$$A(t) = A_s (1 - e^{-\lambda t_{irr}}) \quad (2.8)$$

For a nuclide of the activity A_0 the activity after a decay time t_d becomes

$$A(t) = A_0 e^{-\lambda t_d} \quad (2.9)$$

The number of decays happening in a sample of activity A_0 during a measurement time of t_m can be calculated using equation 2.10.

$$F = \int_0^{t_m} A(t) dt = \frac{1}{\lambda} A_0 (1 - e^{-\lambda t_m}) \quad (2.10)$$

So the number of decays for a given nuclide in a sample after irradiation for t_{irr} and a decay time of t_d registered during a measurement time of t_m can be written as in equation 2.11:

$$F = A_s (1 - e^{-\lambda t_{irr}}) (e^{-\lambda t_d}) \frac{1}{\lambda} (1 - e^{-\lambda t_m}) \quad (2.11)$$

During measurement the number of decays happening at a given energy are collected in the multichannel analyzer, giving a spectrum. As the γ -energy and intensity of the nuclides is known, peaks in the spectrum can be identified and assigned to a certain nuclide. This offers a very simple qualitative analysis.

For quantitative analysis, the above considerations can be used to calculate the mass of a nuclide corresponding to the peak area F as in equation 2.11.

If the saturation activity A_s of a nuclide is known, the saturation activity per mass A_{sg} can be calculated using equation 2.12 and used to calculate the absolute mass of a nuclide by equation 2.13.

$$A_{sg} = \frac{A_s}{NL} = \frac{\phi \sigma}{L} \quad (2.12)$$

$$M = \frac{F}{A_{sg} (1 - e^{-\lambda t_{irr}}) (e^{-\lambda t_d}) \left(\frac{1 - e^{-\lambda t_m}}{\lambda} \right)} \quad (2.13)$$

Using the total mass m of the sample, the concentration of the nuclide contained can be calculated using equation 2.14:

$$C = \frac{F}{A_{sg} (1 - e^{-\lambda t_{irr}}) (e^{-\lambda t_d}) \left(\frac{1 - e^{-\lambda t_m}}{\lambda} \right) m} \quad (2.14)$$

2.4.1 Error calculation

For the calculation of the error of the concentration calculated using equation 2.14 we rewrite the equation using the relation

$$\left(1 - e^{-\lambda t_{irr}} \right) \left(e^{-\lambda t_d} \right) \left(\frac{1 - e^{-\lambda t_m}}{\lambda} \right) m = T_m \quad (2.15)$$

to reach

$$C = \frac{F}{A_{sg} T_m} \quad (2.16)$$

Considering the values t_d , t_{irr} , t_m , and m to be error free, error propagation leads to the error of the concentration σ_C to be:

$$\sigma_C = \sqrt{\left(\frac{\partial C}{\partial F} \right)^2 \sigma_F^2 + \left(\frac{\partial C}{\partial A_{sg}} \right)^2 \sigma_A^2} \quad (2.17)$$

with σ_F the error of the peak area and σ_A the error of the Saturation activity. Naturally, A_{sg} should not have any error, but as its value is derived by experiments rather than calculation from constants, σ_A represents all external errors introduced by neutron flux, detector geometry and cross-sections.

The two partial derivatives are

$$\frac{\partial C}{\partial F} = \frac{1}{A_{sg} T_m} \quad (2.18)$$

and

$$\frac{\partial C}{\partial A_{sg}} = \frac{F}{A_{sg}^2 T_m} \quad (2.19)$$

Inserting equations 2.18 and 2.19 into equation 2.17 the final equation for σ_C becomes equation 2.20:

$$\sigma_C = \frac{1}{A_{sg}^2 T_m} \sqrt{F^2 \sigma_C^2 + A_{sg}^2 \sigma_F^2} \quad (2.20)$$

2.5 Separation of Lines of Similar Energies

Some nuclides have energy lines that lie very close and cannot be separated by common Gauss fitting. The peak created by those nuclides is not noticeably deformed (see figure 3.11 on page 38) and requires a different means of separation.

Considering that the peak area F_i created by the nuclide i follows equation 2.11, the total peak area of two overlapping peaks in a single measurement is

$$F_{tot} = \sum_i F_i \quad (2.21)$$

$$(1 - e^{-\lambda_i t_{irr}}) (e^{-\lambda_i t_d}) \left(\frac{1 - e^{-\lambda_i t_m}}{\lambda_i} \right) = \tilde{t}_i \quad (2.22)$$

Using equation 2.22 we can rewrite equation 2.21 to

$$F_{tot} = \sum_i A_{s_i} \tilde{t}_i \quad (2.23)$$

Considering several spectra k with different \tilde{t}_{ki} we arrive at a system of linear equations:

$$\begin{pmatrix} t_{00} & t_{01} & \cdots & t_{0i} \\ t_{10} & t_{11} & \cdots & t_{1i} \\ \vdots & & \ddots & \\ t_{k0} & t_{k1} & \cdots & t_{ki} \end{pmatrix} \cdot \begin{pmatrix} A_{s_0} \\ A_{s_1} \\ \vdots \\ A_{s_i} \end{pmatrix} = \begin{pmatrix} F_0 \\ F_1 \\ \vdots \\ F_k \end{pmatrix} \quad (2.24)$$

With a system of equations like 2.24, the values for the A_{s_i} can be calculated. In a more simple form, equation 2.25 has to be solved for the vector X :

$$A \cdot X = Y \quad (2.25)$$

The vector Y represents the total peak areas in the measurements and A represents the different time factors.

In the case of two (or more) nuclides, the system is over-determined, so a single solution does not exist. To still get a solution, a least square fit [7, 1] is used.

To improve the accuracy of the solution, the two matrices A and Y are weighted with the inverse of the counting error.

$$\tilde{A}_{ij} = \frac{A_{ij}}{\sigma Y_i} \quad (2.26)$$

$$\tilde{Y}_i = \frac{Y_i}{\sigma Y_i} \quad (2.27)$$

The least squares condition is that equation 2.28 becomes a minimum

$$(\tilde{A} \cdot X - \tilde{Y})(\tilde{A} \cdot X - \tilde{Y})^T \quad (2.28)$$

The corresponding solution for X becomes

$$X = (\tilde{A}^T \cdot \tilde{A})^{-1} \cdot \tilde{A}^T \cdot \tilde{Y} \quad (2.29)$$

The variance of this solution is

$$\sigma x_i = \sqrt{S^2 \left[(\tilde{A}^T \cdot \tilde{A})^{-1} \right]_{ii}} \quad (2.30)$$

with

$$S^2 = \frac{(\tilde{A}^T \cdot \tilde{A})^{-1}}{m - n} \quad (2.31)$$

where m equals the number of equations (spectra) and n the number of variables.

Chapter 3

User's Manual

This chapter can be used as a stand-alone users manual for the Neutron Activation Analyzer. Section 3.1 is a Step-by-step guide for simple measurements and acts as a simple introduction to the system. The following sections offer a detailed description of the hard- and software of the system and all possible options.

3.1 Step-by-step Guide

To perform a simple fully automatic measurement on one sample the following steps are necessary:

1. Physical preparation of the sample material by weighing the sample and filling it into the sample container.
2. Preparation of the header- and script file. For this step the simple batch file `prepare.bat` (see listing 3.1) was created. It is called with two parameters, the first one being a short (three to four letters) name for the sample and the second one being the template header-file for the measurements. A possible call could be

```
prepare spi hd8k4m
```

This call will create the subdirectory `spi` and copy the template header `hd8k4m.dat` into the directory and rename it to `header.dat`. The Nuclear Data program `pars` will then be called to edit the header-file. The most important entry in the header-file is the sample mass; sample name and description are also recommended. The editor can be left by pressing F10.

After the header-file preparation the file called `template.scr` will be copied into the subdirectory and renamed into `spi.scr`. It then will be opened in an editor.

Here the measurement parameters like irradiation time and number of consecutive spectra with their measurement times have to be defined using **GSPEC**'s script language, described in more detail in section 3.4.2. In most cases it suffices to replace all occurrences of the string 'tmp' with the sample name. This editor can be closed by pressing **ALT-D** and **B**. *The file has to be saved!*

3. The sample changer has to be brought into the SHOOT-position. Usually that is the position the sample changer is in.
4. The sample is brought into the sample changer top down.
5. Measurement is started. The most convenient way to do this is to call the batch-file `measprn.bat` (see listing 3.5) with the sample name (e.g. `spi`) as parameter. This batch-file will automatically get the next sample into the SHOOT-position and start the measurement by calling **GSPEC** with the sample name. After the measurement automatic analysis is started by a call to **NDANAEX** and the resulting `result.txt` file will be printed.

Listing 3.1 `prepare.bat`

```
d:
cd smdet      REM change to measurement directory
md %1         REM create directory for sample data
cd ..
cd lemmel     REM change to directory containing default header
copy %2.dat d:\smdet\%1\header.dat REM copy default header
pars d:\smdet\%1\header.dat      REM edit header data
cd \smdet\%1
copy \lemmel\template.scr %1.scr  REM copy script template
c:\dos622\edit %1.scr             REM edit script
cd \lemmel
```

3.2 Components/setup

The Neutron Activation Analyzer has to fulfill three major functions. It takes care of *irradiation*, *measurement*, and *analysis*. To this end all the



Figure 3.1: Front view of the measurement position. At the top the end of the plastic pneumatics tube can be seen entering the rabbit separation chamber. In the center of the big aluminum ring rests the detector head. The two plastic tubes at the bottom are for sample ejection and air flow respectively from left to right.

mechanical and electronic components have to be controlled by one central computer.

This common PC controls the pneumatics to move the sample between the irradiation position and the measurement position. The PC also has full control over the sample changer.

The program **GSPEC** takes care of irradiation and data collection from the detector. After the collected spectra are saved to disc the program **NDANALYZ** performs the automatic peak evaluation and calculates the concentrations of the found nuclides.

3.2.1 Pneumatics

The main goal of the pneumatics system is to propel the sample into the neutron flux in the reactor core and back out after irradiation time to the

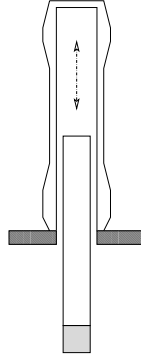


Figure 3.2: The sample-rabbit separation process.

detector. The transportation time should be as short as possible to have the possibility to measure short-lived radionuclides.

A very important aspect is that both positions, the one in the core and the position in front of the detector have to be the same for every single measurement. Otherwise neither neutron-flux nor detection geometry would be reproducible and no significant measurements could be made.

To insure correct sample position within the reactor core, the sample contained in a rabbit falls into the slightly conical end of an aluminum tube. The rabbit fits the end of the tube snugly and so the position within the core cannot vary.

The rabbit also provides a simple measure to prevent contamination of the sample by aluminum dust. To this end the sample must leave the rabbit right before entering the measurement position (as seen in figure 3.1).

The sample – rabbit separation is inertia driven. Once the rabbit (including the sample) enters the separation chamber, the rabbit connects with the floor of the chamber which has a hole with the diameter of the sample in it. The rabbit stops and the sample keeps falling downwards into the measurement position as seen in figure 3.2.

3.2.2 Sample Changer

The sample changer as seen in figure 3.3 consists mainly of a large plexiglass cylinder with an eccentrically placed hole the size of the sample.

During one cycle the main cylinder makes one full rotation, stopping at three positions:

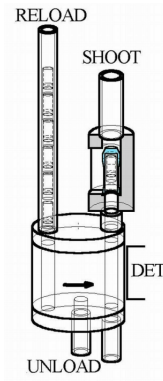


Figure 3.3: The sample changer.

Shoot This is the position right next to the detector, beneath the sample-separation chamber. In this position the sample can be shot into the reactor and upon arrival be measured. This is the main position.

Unload When the cylinder stops at this position the sample container falls through a hole in the bottom aluminum layer and slides into a lead container.

Reload Upon arrival in this position two electro magnets are powered and allow one (new) sample to drop into the sample changer from the sample stack. The top magnet holds the next-to-last sample in place, while the lower magnet lets the last sample drop. As soon as the cylinder starts moving again, the two magnets loose power, i.e. the lower magnet closes the sample tube and the top magnet lets the next sample move one position down. The stack can contain up to ten samples and consists of an aluminum tube.

After fetching the next sample from the **RELOAD** position, the sample changer moves to the position **SHOOT** and the next measurement can be started.

3.2.3 Electronics

For safety reasons the Neutron Activation Analyzer works not only in fully automatic mode but also in manual mode, where each component can be controlled by hand. For this reason and to act as interfaces to the PC, four circuits have been developed:

- the internal PC-Card for data collection with loss free counting module
- the controller for the sample changer
- the separate controller for the pneumatics
- a central decoding unit to stop misfiring of either pneumatics or sample changer and to allow manual control

Pneumatics controller

The circuit board to control the pneumatics has been developed separately as it contains high voltage relays (230 V). Its main function is to provide control of the relays to the PC via the decoding unit and to open the valves only for short bursts in manual mode.

Sample changer controls

The circuitry for the sample changer can be found in figure 3.4.

The switches S1 and S6 are for the power supply for the motor and the magnets respectively. Switches S2, S3, and S4 are microswitches, mounted in contact with the plexiglas cylinder. Each switch stops the cylinder at one of the three positions RELOAD, SHOOT, and UNLOAD by triggering the RELAY. At the same time the three LEDs LED1, LED2, and LED3 light up to show the current position of the sample.

In position SHOOT the optocoupler OK2 powers pin PC-PD2 which is connected to the main PC.

In position RELOAD the two magnets are triggered to drop the next sample into the cylinder.

Switch S5 provides manual control. If pressed, the RELAY triggers and the motor moves to the next position. The same function offers pin PC-PP. Again this pin is connected to the PC (via the decoding interface) and if put on high the cylinder moves to the next position.

Decoding unit

The circuitry for the decoding unit can be found in figure 3.5.

The decoding unit uses three DIP-switches and three logic comparators to decode three different 8-bit signals. If the 8-bit signal coming from the PC corresponds to the code on the DIP-switch, the corresponding pin is set to high. pin MV is connected to pin PC-PP in the sample changer circuitry (figure 3.4) and pins SI and SO are connected to their corresponding pins in the pneumatic controller to open and close the valves.

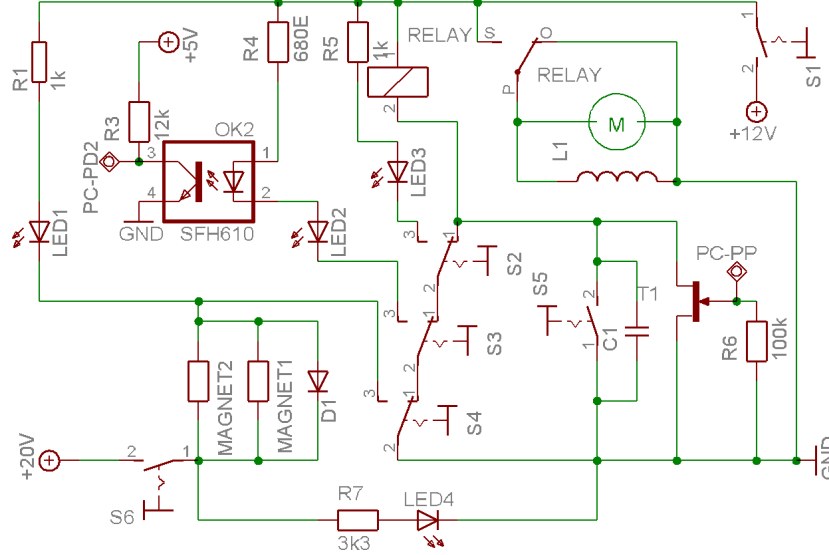


Figure 3.4: Circuitry for the sample changer.

The decoding unit became necessary because, especially during booting, the parallel port of the PC is not in a defined state. This caused randomly opening valves and moving sample changer. The probability of the 8 pins on the parallel port of the PC to be in the exact state necessary to pass the decoding unit is negligible, this has not happened yet.

3.3 Software

The Neutron Activation Analyzer needs a multichannel analyzer that can handle the correction factors produced by the Loss Free Counting module and some analyzer software that finds peaks in spectra, identifies those peaks, and uses the additional information in the corrected spectra to calculate concentrations from peak areas.

To this end Hartmut Lemmel programmed the multichannel analyzer GSPEC and the peak analysis software NDANALYZ [4]. Both programs use the Nuclear Data file format. Lemmel also built several utilities to ease conversion and handling of spectral data files. Later Christoph Bloch extended the functionality of NDANALYZ [1] to include separation of coinciding lines of different nuclides, renaming the program to NDANAEX.

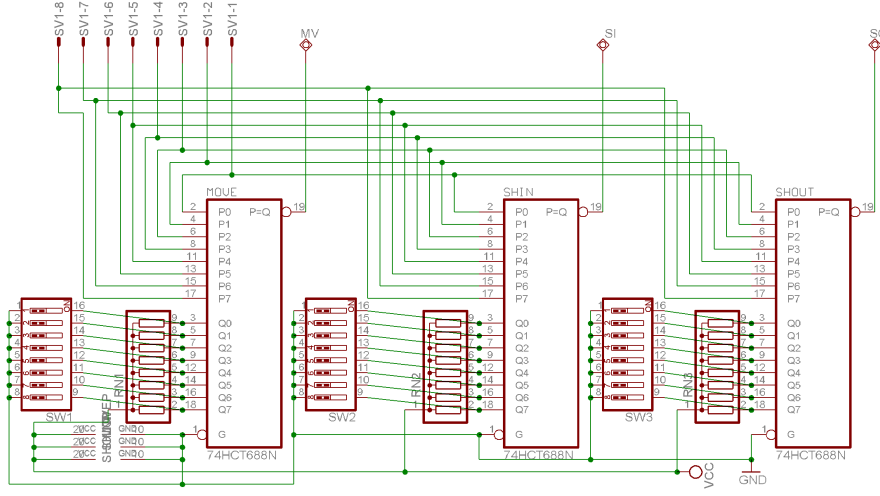


Figure 3.5: Circuitry for the central decoding unit.

3.3.1 GSPEC

The program **GSPEC** is in itself a multichannel analyzer, that uses the correction factors produced by the Loss Free Counting module to produce spectra.

GSPEC runs under DOS to have full control of interfaces and memory. It is written mainly in C++ and uses machine code for parts where speed is vital.

The program calculates additional correction factors from a look-up table to correct for pulse pileup. This calculation has to work in real time.

To utilize the full potential of Loss Free Counting, **GSPEC** produces several pairs of spectra for a single measurement. Each pair consists of an uncorrected and a corrected spectrum. **GSPEC** provides the ability to seamlessly count several spectra, each representing a different slot of measurement time. Using those spectra it becomes possible to easily separate long and short lived nuclides. To simplify this separation there must not be any time between two connected pairs of spectra.

To be able to work on so many spectra in real time and not loose any information during disk access, all spectra are saved into high memory. Only after the last spectrum is saved and the measurement is finished, all data are written to disk.

GSPEC works in two different modes, either in script mode, where a script file containing measurement data is specified (see section 3.4.2), or in manual mode where the user can control every aspect of the measurement cycle from the user interface (see section 3.4.1). The program also controls the sample

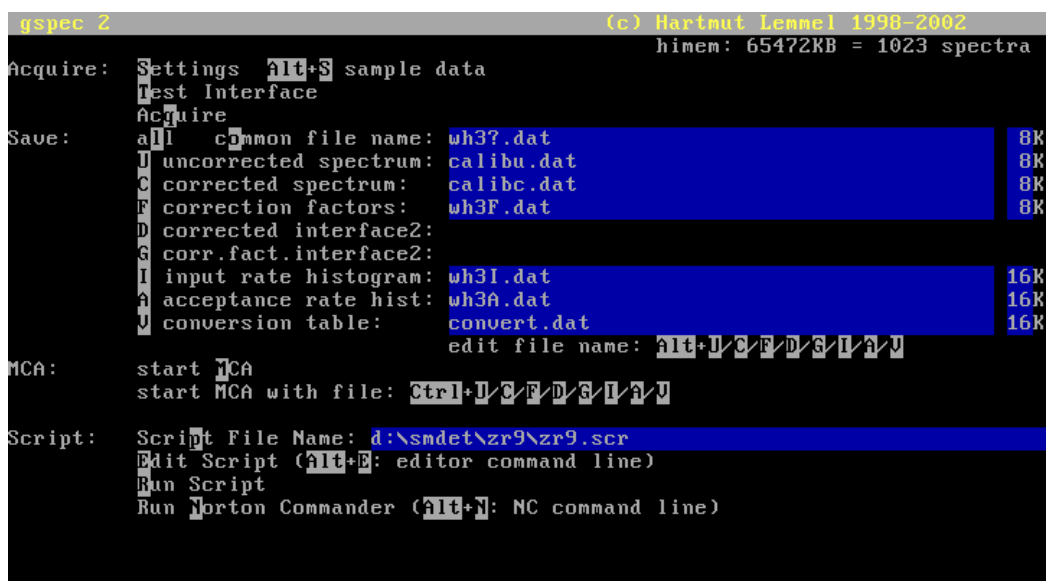


Figure 3.6: Main screen of the program GSPEC.

changer and the pneumatics, again either via a script file or from manual input.

Main Menu

The main menu of GSPEC can be found in figure 3.6 and is the first view the user gets after starting GSPEC.

In general all options on any screen in GSPEC are explained on the same screen. To access fields or features the grey marked letters are pressed.

The main screen is divided into four parts:

Acquire All necessary options for manual acquisition of spectral data. The corresponding features and options will be described in more detail further down.

Save Filenames for different output files can be entered into the fields by pressing the marked key and the ALT key together. Spectral resolution can also be entered here. By pressing the corresponding key without ALT the file is saved to disc.

MCA Starts the Nuclear Data Multichannel-analyzer MCA with or without spectral data.

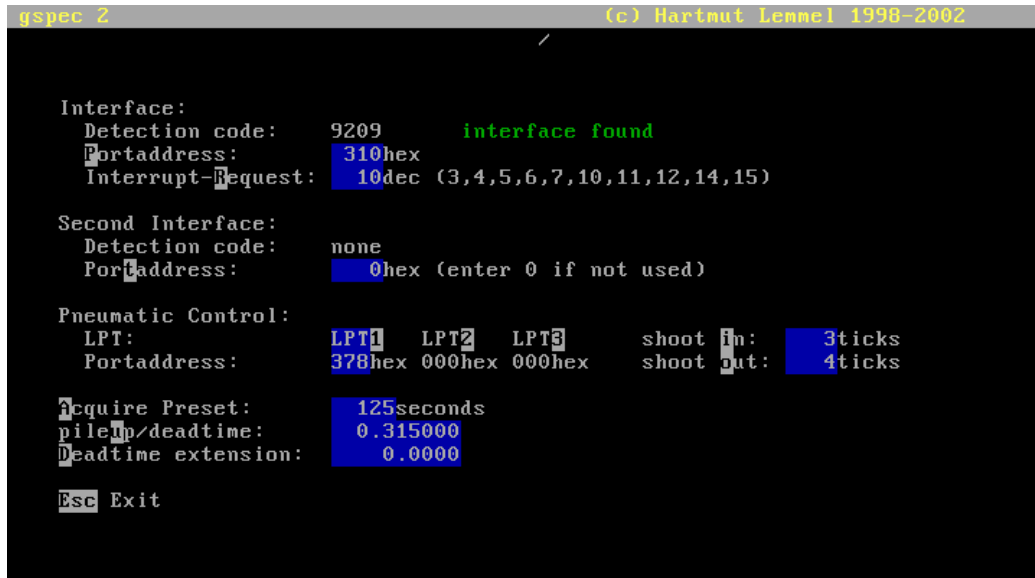


Figure 3.7: GSPEC Settings screen

Script Offers control over scripts. A script name can be entered, the corresponding file edited with two different editors and run.

Settings

The Settings Screen (see figure 3.7) can be reached from the main screen by pressing **s**, and controls several important settings for acquisition. It allows to enter port addresses for one or two interface cards as well as to control the pneumatics interface. Pneumatics are controlled through one of the parallel ports of the PC and the time for shoot in and shoot out can be entered. Those values will only be accessed from the *Test Screen*.

The time value in the *Acquire preset* field sets the time for manual acquisition using the *Acquire Screen*.

The last two values are important parameters for Loss Free Counting. The dead-time extension should be set to zero if the interface card used produces dead-time weighing factors and pulse-pileup correction is to be calculated from a lookup table. The fraction pileup/deadtime controls this calculation (see section 2.3).

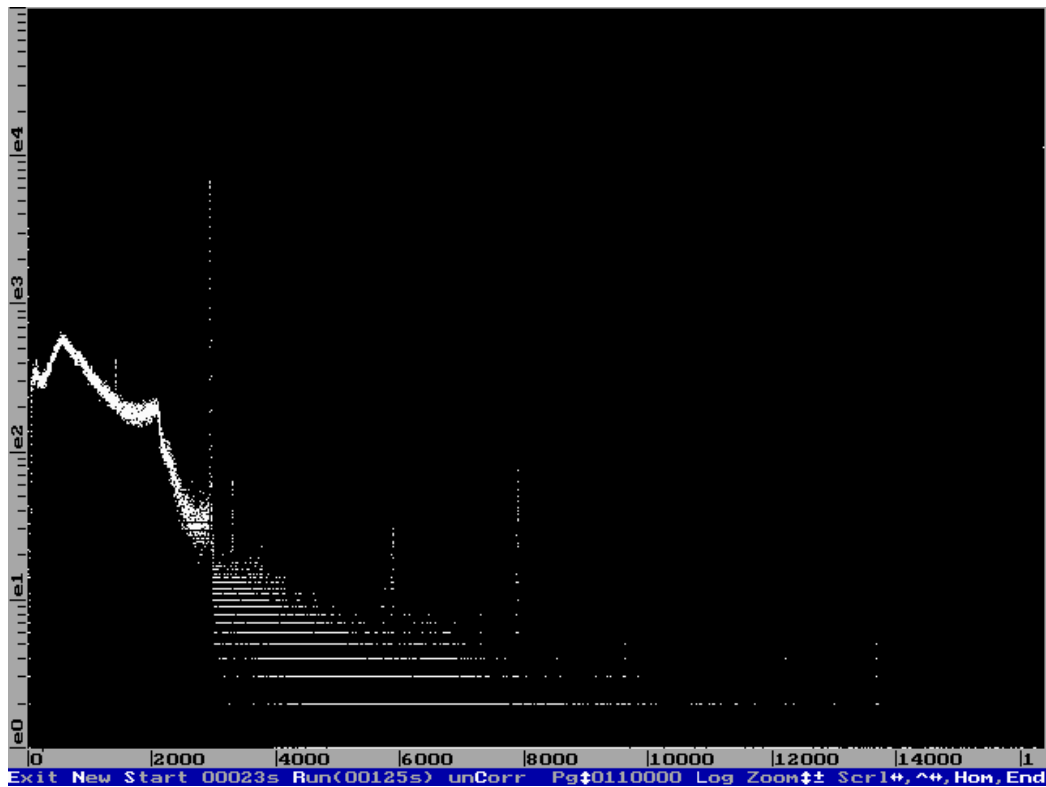


Figure 3.8: GSPEC Acquire screen

Acquire Mode

Acquire Mode (see figure 3.8) can be reached by pressing **q** in the main screen. It offers simple control over the spectral data shown on screen.

To clean the screen and all spectral data in memory the key **n** for New is pressed. With **Start** the acquisition is started and can be stopped by pressing **s** again. For a timed measurement the option Run is used. This will start acquisition and stop after the indicated time which can be changed in the settings screen as described on page 31.

Using the option unCorr, different spectral data can be viewed, showing either the corrected or the uncorrected spectrum as well as several histograms.

The options on the left hand side are self-explanatory and offer different views of the spectral data.

It is important to note that the data acquired (or deleted) in Acquire Mode can then be saved to disc by using the main menu.

```

gspec 2                                     (c) Hartmut Lennel 1998-2002
Test Interface -
Data Corr
value: 08E4h 0171h
min: 0000h 0100h
max: FFFCh 051Bh

acceptance rate: 41542 cps (avg. in 32 ticks)
primary corr.factor: 1.40 given by the interface
input rate: 58245 cps (avg. in 32 ticks)
final corr.factor: 1.58 software corrected
correction rate: 65661 cps (avg. in 32 ticks)
total counts: 890514
time: 00032 sec
evaluate specC/specU:

flags: bit8=1 Interrupt enabled loops
bit9=0 Interrupt processed read data

Test pneumatic control: reset, shoot in load flags=78
shoot out pos=0

Press Esc to exit
Press 0 to reset min/max, press h to hold the display
position:

```

Figure 3.9: GSPEC Test Interface screen

Test Interface

The Test Interface screen as seen in figure 3.9 offers a quick overview over the activity of the interface. The current count-, acceptance-, and input-rate can be found on the screen along with the corresponding correction factors.

At the bottom, controls for the sample changer and the pneumatics can be found.

Measurement

The Measurement Screen (figure 3.10) is shown during automated measurements using script files.

At the top the path and name of the script-file can be found. Below the current steps in the script file are shown along with the information on how long the current step and the total measurement will be running. this information is given in ticks as well as in seconds.

At the bottom of the screen the filenames for out- and input-files can be found.

Logging

GSPEC logs all measurements into two separate files. Firstly the log-file provided in the script command and secondly a continuous log-file in the subdi-

```

gspec 2                                     (c) Hartmut Lennel 1998-2002
d:\smdet\nca\nca.scr                      prep: 00300000h
  Interface: data1=0524h flag8=1          shoot in: 3 ticks  histacc= 1*00160000h
              data2=01CEh flag9=0        shoot out: 4 ticks  histinp= 1*00170000h
                                              corrconv=00100000h

      Step:      1    of      13 (Irradiation)
      Mode:
      Filename:
      Spectrum:   -    of      24
      Time:       23    of     182 Timer Ticks, approx.    1    of     10 Seconds
      Schedule:   0 till 182 Timer Ticks, approx.    0 till 10 Seconds
      Total Time: 23    of    4016 Timer Ticks, approx.    1    of    221 Seconds

log file: d:\smdet\nca\test.log
acceptance rate histogram: ncaacc#.dat
input rate histogram: ncainp#.dat
template file: d:\smdet\nca\header.dat

The measurement is running.
Press Esc to abort.

```

Figure 3.10: GSPEC Measurement screen, shown during measurement

rectory GSPEC is located. This continuous file is named yyyy-mm.log, where yyyy stands for the year and mm for the month. This feature is especially useful when looking for the last irradiation of a given sample.

A sample log-file can be found in listing 3.2.

3.3.2 NDANALYZ

The program NDANALYZ provides a very sophisticated front-end to the Nuclear Data program PEAK. NDANALYZ was explicitly written for analysis of spectra collected by GSPEC.

To call NDANALYZ, several command-line parameters have to be included. A usual call would probably look like:

```
ndanalyze bcr###?.dat *.pk satur.txt result.txt detail.txt
```

bcr###?.dat Mask for the filenames for the spectra, where the # denotes a number and the ? stands for the u or c of the uncorrected or corrected data. (e.g. bcr001c.dat or bcr010u.dat)

***.pk** Mask for the output filenames containing the analysis of two corresponding single spectra.

satur.txt Name of the library-file containing the saturation data.

Listing 3.2 Example log-file, a clipping from the file 2004-06.log

```
log file d:\smdet\A60\test.log created by gspec.exe
Date: 2004/06/07
Time: 09:59:15
Script: \smdet\A60\A60.scr
Sample data:
  Title   =Rohrpost IAEA      8 k channels      4 MeV full scale
  Id      =A60
  Type    =AudiA6100000km
  Quantity=1.000000 g

time measurement in "ticks" = in cycles of the timer interrupt
1 tick = 1/18.2 s (exact: 65536/1193180)

starttick ticks---- startmsec millisecs saved as {uncorr,corr,factor,corr2,fact2}
000000000 000032772 000000000 001800018 {d:\smdet\A60\A60001U.dat,d:\smdet\A60\A60001C.dat,,}
save: 001B0000h histogram {d:\smdet\A60\A60acc0.dat}
save: 001C0000h histogram {d:\smdet\A60\A60acc1.dat}
save: 001D0000h histogram {d:\smdet\A60\A60acc2.dat}
save: 001E0000h histogram {d:\smdet\A60\A60inp0.dat}
save: 001F0000h histogram {d:\smdet\A60\A60inp1.dat}
save: 00200000h histogram {d:\smdet\A60\A60inp2.dat}

measurement finished
```

result.txt Name of the file containing the final results of the analysis (optional, **result.txt** is default).

detail.txt Name of the file containing the detailed final analysis (optional, **detail.txt** is default).

The above call would tell NDANALYZ to look for all spectral data files corresponding to the filename **bcr###?.dat**, starting at **bcr000c.dat** and **bcr000u.dat** in the current directory. NDANALYZ will automatically only read files that exist and skip missing numbers.

The program then uses PEAK to look for peaks in the spectral data files and compare the peaks found with the given energies in the library-file. If NDANALYZ finds a matching peak, it will use the given saturation A_{sg} in the library-file and the peak area from the corrected spectral data file F to calculate the concentration of the nuclide, using equation 2.14 (see chapter 2.4). All the other necessary information (t_{irr} , t_d , t_m , m) NDANALYZ reads from the header of the spectral data file.

The concentrations of all found nuclides in two corresponding spectra (corrected and uncorrected) will be saved to the file ***.pk**.

After calculating all the concentrations in all the files, NDANALYZ will go through all nuclides found again, calculate the weighted mean of the concentrations found and save these into the files **result.txt** and **detail.txt**. The weight used for this calculation is the inverse of the square of the corresponding error.

A typical result given can be found in listing 3.3

Listing 3.3 Example output of NDANALYZ

```
result.txt
Tir= 10s
Weight= 3.58g
```

	E(lib)	E(found)	conc	err%	items
Sc-46m	142.530	143.310	4.924e-07	8.663e+00	3
Ba-139	165.865	164.127	1.352e-02	4.156e-02	3
O-19/F	197.140	197.970	4.273e-05	1.144e+02	1
O-19a	197.140	197.970	4.352e-01	8.644e+01	1
Ti-51	320.077	321.015	1.630e-04	4.815e+01	2
I-128	442.900	443.980	3.420e-06	7.463e+01	1
Rb-86m	556.020	556.990	1.765e-05	4.198e+01	2
Br-80g	616.350	618.115	2.308e-05	9.741e+00	2
Cl-38m	671.300	672.993	2.124e-03	1.180e+00	3
Mn-56a	846.600	846.553	6.537e-05	2.328e-01	3
Mg-27	1014.400	1015.175	1.283e-03	2.263e+01	2
Cu-66	1039.200	1039.900	2.552e-05	4.528e+01	2
Na-24a	1368.600	1368.570	1.928e-04	9.611e-01	1
V-52	1434.060	1433.903	5.632e-06	5.422e+00	3
K-42	1524.700	1524.550	2.252e-03	2.092e+00	1
Cl-38a	1642.300	1642.277	1.204e-03	2.079e+00	3
P-31/Al-28	1778.850	1778.264	6.500e-01	1.924e-01	5
Al-28	1778.850	1778.264	9.596e-04	6.430e-01	5
Mn-56b	1811.200	1809.570	5.411e-05	8.644e-01	2
Cl-38b	2167.400	2165.667	2.023e-03	7.011e-01	3

3.3.3 NDANAEX

During April and May 2004 Christoph Bloch extended the functionality of NDANALYZ [13, 1] to include the possibility to separate lines of different nuclides at almost the same energy like, for example $^{116\text{m}}\text{In}$ and $^{77\text{m}}\text{Se}$, both at 162 keV. NDANAEX has all the same command line parameters that NDANALYZ has and should be used instead.

To separate two peaks that lie on top of each other and cannot be separated by Gauss fitting (see figure 3.11) a least-square fit [7] is implemented. For more information on the algorithms used, see section 2.5.

NDANAEX works in three steps:

Search library: NDANAEX looks through the library to find lines that fulfill the following conditions:

1. $|E_1 - E_2| < 2\Delta E$
2. $\lambda_1 \neq \lambda_2$
3. $A_{sg_i} \neq 0$; $\lambda_i \neq 0$

where ΔE gives an empirical guess at the ability of the system to separate lines.

The program recognizes two special cases:

1. For two lines with similar energies but the *same* half life a separation is not possible and the program stops after giving an error message.
2. If several lines have the *exact* same energy and the same half life, only the line coming last in the library will be used.

Creating a list of possible peaks: While the spectrum is analyzed and lines are identified, the program watches for lines that may need to be separated and saves the peak data of the corresponding line in a list.

Weighted Least Square Fit [7]: After all the spectra have been analyzed and the above list completed, a weighted least square fit is used on the lines found and the corrected concentrations are put into the result file into a separate column.

3.3.4 Other Utilities

There are several other utilities for easy manipulation of header files, spectral data files or output files.

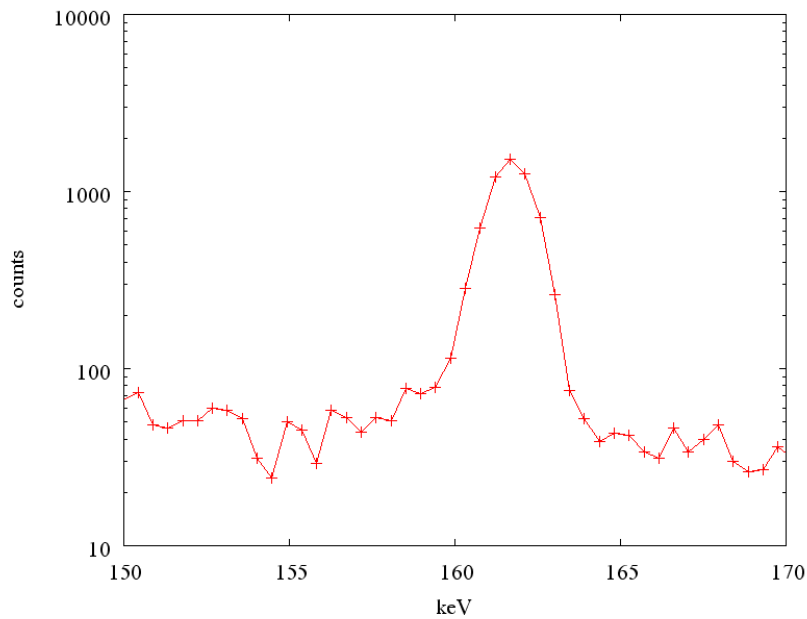


Figure 3.11: Inseparable peak of $^{116\text{m}}\text{In}$ and $^{77\text{m}}\text{Se}$

NDHEADER

NDHEADER copies *all* the header data from one spectral data file to one or more other files.

NDSUM

NDSUM offers the possibility to add several spectra and create a new one. The syntax is for example:

```
ndsum spi###?.dat spi100?.dat 1 99
```

This command will sum the files `spi001c.dat` to `spi099c.dat` into the file `spi100c.dat` and do the same for the corresponding uncorrected files.

NDCAL

The program NDCAL is a slight variation of NDHEADER and copies only the energy calibration data from one spectral data file to one or more others. This became necessary to simplify the task of changing energy calibration after measurement for several tens of files without the tedious task of manually changing the data for every single file.

NDCALS

NDCALS copies not only the energy calibration but also the sample data from one file to one or more others.

READPK

This program needs the *.pk files produced by NDANAEX or NDANALYZ and searches them for the occurrence of a certain nuclide. All lines starting with the given string are read and written into the output file in a slightly different order. The call

```
readpk zr0 10 zr.out Zr-90m
```

will search the files zr0001_.pk to zr0010_.pk for the string Zr-90m and copy all occurrences into the file zr.out.

PARS

The program PARS by Nuclear Data is an editor for the header data of a Nuclear Data format spectral data file.

3.4 Measurements

The Neutron Activation Analyzer offers several ways to make measurements. The most convenient way is the fully automatic mode described in 3.4.3, but there is also the possibility to use the system as a simple multichannel analyzer with loss free counting.

3.4.1 Manual (no automatic analysis/within GSPEC)

GSPEC allows manual measurements using only its menu capabilities. After starting GSPEC the main menu as seen in figure 3.6 is shown.

Pressing q starts the *ACQUIRE* mode. The monitor shows a spectrum containing old data. The x-axis denotes the number of channels and the y-axis the number of counts. The bottom line shows a menu offering several options.

3.4.2 Semi-automatic (using scripts)

A very comfortable way to use GSPEC is its ability to read and use script files. A script file contains all the necessary information for GSPEC to complete a measurement. A sample script file can be found in listing 3.4.

There are two ways to use **GSPEC** with a script file. The name of the script file can be entered in the corresponding field in the main screen (see figure 3.6) and then the script can be started with the command **Run script** by pressing **r**. After pressing enter the script will start.

To use **GSPEC** in a more automatic way, the name of the script file can be given as command line parameter at start of the program. This way, **GSPEC** automatically loads the script file and starts to run. After the script file is finished, **GSPEC** stops and returns to **DOS**. That way, several measurements can be automatically performed with a simple batch-file (see section 3.4.3).

Script Commands

In general script, files contain some information about the sample (i.e. which file contains the header), some information about the data that should be collected (spectral data file names, histograms) and some timing information (irradiation time, measurement time).

Time periods can be specified either in seconds with an **s** after the number or in *ticks* with a **t** after the number. One second is about 18.2 ticks long ($1\text{ s} = \frac{1193180}{65536}\text{ ticks}$). **GSPEC** internally uses ticks, all periods given in seconds will be rounded to full ticks.

The scripting language allows comments in the script file, which **GSPEC** will ignore. A comment can be started with a semicolon and ends at the end of the line.

GSPEC understands several commands in script files:

setpath Sets the path for the input and output directory

settemplate Sets the filename for the header file, which will be used as a template for the following measurements

setlog Sets the filename of the log file

setsize Sets the size of the spectral data files (8, 16 or 32 kb)

saveacceptrate Sets the filename for the acceptance rate histogram

saveinputrate Sets the filename for the input rate histogram

loadsample Loads the next sample from the sample changer

setshootin Sets the time constant for the opening of the shoot-in valve

setshootout Sets the time constant for the opening of the shoot-out valve

Data types for acquisition	
Data type	Number
uncorrected	1
corrected	2
correction factors histogram	4

Table 3.1: Data types for the `acquire` command

irradiate Sets the time for the irradiation (the time between shoot-in and shoot-out)

acquire Tells GSPEC to acquire a single spectrum. `acquire` needs several arguments: `acquire time type name` where `time` is the measurement time (collection time), `type` the kind of spectral data to collect and `name` the name (template) of the output file.

The type is a number defining which types of spectra to acquire. For multiple types of spectra the numbers are added. The different types can be found in table 3.1.

repeat # to # — endrep These two commands offer the possibility to create simple loops. All the commands between `repeat` and `endrep` will be repeated as many times as specified. The number of repetition can be accessed with the `#` sign. The usual use for this loop is the collection of several spectra with equal measurement time, for example

```
repeat 1 to 10
  acquire 18t 3 bcc###?.dat
endrep
```

will collect 10 uncorrected spectra (type 1) with 18 ticks (1 second) measurement time each into the files `bcc001u.dat` to `bcc010u.dat` and the ten corresponding corrected spectra (type 2) into the files `bcc001c.dat` to `bcc010c.dat`.

Loops can be nested, but only the innermost counter can be accessed.

3.4.3 Fully Automatic (including analysis)

To perform fully automatic analysis with the Neutron Activation Analyzer is probably the best and easiest way to process many samples in little time

Listing 3.4 Example of a script file for use with GSPEC

```
setpath d:\rubbit\bbf\      ;sets the path for measurement data
settemplate d:\rubbit\bbf\header.dat ;specifies the header template
setlog test                 ;sets the log-file name
setsize 8                   ;spectrum size (8K channels)
saveacceptrate bbfaccc.dat  ;histogram information
saveinputrate bbfinp.dat

;loadsample

setshootin 3                ;shoot-in time
setshootout 4               ;shoot-out time
irradiate 182t              ;irradiation time (in ticks)

repeat 0 to 0               ;repeat once
  acquire 18t 3 flight?.dat ;acquire a pair of corr. and uncorr.
endrep                      ;spectra , 18 ticks long
repeat 1 to 1
  acquire 36t 3 bbf###?.dat
endrep
repeat 2 to 2
  acquire 364t 3 bbf###?.dat
endrep
repeat 3 to 3
  acquire 3640t 3 bbf###?.dat
endrep
repeat 4 to 4
  acquire 18200t 3 bbf###?.dat
endrep
;repeat 100 to 119
  ;acquire 2184t 3 bbf###?.dat
;endrep
```

with full results in between measurements. In fact fully automatic analysis is simply a matter of using a DOS batch file to perform all necessary tasks in the correct order without time loss.

The simplest batch file for automatic analysis performs a *sample change*, starts GSPEC with the corresponding *script file* (see section 3.4.2), performs *automatic analysis* with NDANALYZE (section 3.3.2) right after the end of the GSPEC session and (optionally) prints out the `result.txt` file. Calling this batch file repeatedly for different samples performs automatic analysis without time loss.

The main batch file used for standard measurements can be found in listing 3.5. This file uses the DOS command SHIFT to process multiple entries in the command line.

Listing 3.5 Standard batch file for automatic analysis

```
@echo off
: anfang
if "%1"==" " goto end
next
gspecacq \smdet\%1\%1.scr
cd \smdet\%1
ndanalyz %1###?.dat %1*.pk \lemmel\satur.txt result.txt detail.txt
print result.txt
cd\lemmel
SHIFT
goto anfang
: end
```

3.4.4 Further Possibilities (multiple measurements. . .)

Obviously, all of the above methods of performing a measurement can be combined. Using batch-files, several measurements can be performed subsequently, with or without automatic analysis in between.

Sometimes, when time is an important factor, collecting as many spectra in the given time (neutron source up-time) is more important than instant results, so analysis can be performed after several samples have been measured on all of the collected data. At this time, the PC can be disconnected from the detector system and, as it is mounted on a trolley, relocated at a more convenient position.

Using script files, it is also possible to perform subsequent irradiation with GSPEC automatically adjusting the header information. Thus it is possible to

use only a short radiation time to activate very short-lived nuclides and after taking several spectra, re-irradiate the sample for a longer time to activate long-lived nuclides. By using dummy names for spectral data files, even exactly timed decay after radiation is possible, to get rid of any background radiation from already measured short-lived nuclides.

Including some of the additional tools mentioned in section 3.3.4, namely READPK, in the batch-file performing the measurement offers an easy way to instantly extract interesting information without having to wade through several pages or files.

3.5 Library

Probably the most important data-file used is the library file containing all the necessary information for NDANAEX to perform exact analysis. An abridged sample (omitting unnecessary columns) of the library file currently used can be found in listing 3.6.

Listing 3.6 An abridged sample of the library file `satur.txt`. The columns 5–7 have been omitted for layout reasons.

Col. 1 Nuclide a first Line b second L.	Col. 2 g-line energy keV	Col. 3 Halflife in seconds	Col. 4 GAMMA Intensity absolute	Col. 8 New System Geometry C. cts/g found	Col. 9 Error from U-file %
Na-24b	2754	5,41E+04	1	7,56E+07	4,73
Na-24a	1368,6	5,41E+04	1	1,54E+08	6,2
Ne23/Na	439,9	3,72E+01	0,329	2,69E+05	0,65
Mg-27b	1014,4	5,68E+02	0,28	4,01E+05	3,62
Mg-27a	843,8	5,68E+02	0,72	1,24E+06	9,6
Al-28	1778,9	1,35E+02	1	5,45E+07	12,9
P31Al28	1778,9	1,35E+02	1	1,19E+05	0,68
Si/Al29	1273,3	3,94E+02	0,907	5,09E+05	2,38
S-37	3103,4	3,03E+02	0,941	4,11E+03	5,66
Cl-38b	2167,4	2,23E+03	0,424	5,45E+06	2,17
Cl-38a	1642,3	2,23E+03	0,328	5,28E+06	3,61

The information contained in the library file consists of:

Column 1 A string designating the nuclide and line used. The string must not be longer than 7 characters. The usual designation for nuclides with several lines is to add the letters ‘a’ and ‘b’ to the names of the nuclide. For example the 2754 keV line of ^{24}Na is called **Na-24b** and the line at 1368.6 keV is called **Na-24a**.

Column 2 The energy of the line in keV, for compatibility reasons, the comma is used as the decimal marker, so instead of writing 1368.6, the German version 1368,6 is used.

Column 3 The half-life of the given line in seconds.

Column 4 The absolute γ -intensity. This column and the following columns up to column 7 are not used by `NDANAEX` but contained in the library file for historic reasons and to make calculations easier.

Column 5–7 Here the original data from the Erdtmann tables [2] and some recalculations for the TRIGA-reactor are included.

Column 8 The saturation activity per mass given in cts/g for calculation of concentrations (see equation 2.13 on page 19, A_{sg}).

Column 9 The error of the value in column 8 in percent. This error also includes the error of the given detector geometry, the error of the neutron flux and any other eventual errors.

The columns in the library file have to be separated by tabulators. As the DOS-native editor `edit` cannot read or save tabulators, it must *never* be used to edit the library file. Either a DOS-editor that understands tabulators has to be used or, as is a lot more convenient, a dedicated spreadsheet program.

3.6 Interpretation/Caveats

3.6.1 Input Values

In the end, even a sophisticated system like the Neutron Activation Analyzer is not intelligent by itself. The old *GIGO* (Garbage In, Garbage Out) rule applies, i.e. if any of the data put into the system, either via the samples themselves or any of the input files, is in any way corrupt or meaningless, the final result will be meaningless, too.

A typical example of insensible input via the sample would be proportionally very long or very short irradiation times. If the input rate during data collection rises above 1 million counts per second, the system cannot handle the incoming pulses and leaves all the spectra empty. At the same time, for very short irradiation times, the spectra might turn out empty, especially if the counting times are short, too.

Another typical source of errors is the neutron source. Correct analysis of any given sample relies on constant neutron flux. If, for some reason, the reactor used as source scrams during irradiation or runs at different power levels (in the case of the TRIGA at the Atominstitut, more or less than 250 kW), the results will be severely flawed.

In case of the library-file, a wrong value for any of the important values (see section 3.5) will result in wrong final results. In this case, an error may be very hard to find!

The errors offered in the final result of the program are not only dependent on the errors of the measurements themselves, but also on the error value contained in the library file. Naturally, the error of a given measurement is not only described by the error of the constituent measurements, but also by simple error propagation, by all the errors of the values used to calculate the final value (see section 2.4.1, equation 2.20). Thus the value given in column 9 of the library file (see section 3.5) has to be carefully chosen.

Usually as a starting value, while entering a new nuclide into the library, the most sensible value is zero, as no error can be given. After several measurements with samples of known composition, comparing individual results will lead to a first estimate of the error. Whenever a sample is measured that contains a known quantity of the relevant nuclide, the result should be used to refine both error and saturation value.

3.6.2 Output Values

The results output by the Neutron Activation Analyzer need to be carefully interpreted. When looking for a certain element within a sample, the corresponding nuclide may have several lines. For each of those lines, concentration will be calculated separately. Naturally, the results will not be exactly the same. Especially with lines with different half-lives like, for example ^{38}Cl and $^{38\text{m}}\text{Cl}$, the results may vary. Taking the mean value of the different results presented will in most cases be a very good way to obtain a final result, but a look at the corresponding errors of the values might be in order. Considering that automatic analysis only ‘knows’ what is loaded via the library-file, overlapping peaks, especially peaks overlapping single- or double-escape peaks of other nuclides will not be separated without the necessary information in the library file.

So, if the final output of NDANAEX contains several values for a single nuclide, their respective errors should be taken into consideration.

If the final output shows only one occurrence of a nuclide that actually has more than one line in the library-file, it is possible that energy calibration (in the header-file) has slipped.

Experience shows that, as most natural samples contain either sulphur or calcium, if neither the 3084.4 keV line of ^{49}Ca nor the 3103.4 keV line of ^{37}S are found, energy calibration has been slipping. Especially when restarting the system and re-cooling the detector after a longer period of the detector being warm, calibration has to be reassessed. The easiest way to do this is to measure a known sample, if possible together with ^{241}Am and ^{60}Co sources and use the program GRECAL by nuclear data to recalculate the new values for calibration.

Chapter 4

Evaluation

In general the Neutron Activation Analyzer offers a wide range of different possibilities. It can be used as a simple γ -spectroscope, measuring samples that have been radiated in the dry tubes of the reactor. Exploiting all its different options it can be used as an automatic and fast multi-element analyzer.

The Neutron Activation Analyzer is specialized for short term and high rate γ -spectroscopy, it can process up to one million counts per second and with a transportation time of 300 ms can find nuclides with half lives in the range of several 100 ms.

4.1 System performance

To evaluate system performance, several especially designed experiments, as well as constant observation during long periods of ordinary measurements were performed to test the limits of the system.

4.1.1 Mechanics

A very important part of the stability and usability of any system is the reliability of the mechanics in use. In case of the Neutron Activation Analyzer, the sample changer and the pneumatics have to perform flawlessly and reproducibly every time.

Pneumatics

Most important of the pneumatics system is the reproducibility of transport time. Without a very short and standardized transport time, the first spectrum of each measurement can be severely flawed. Considering short-lived

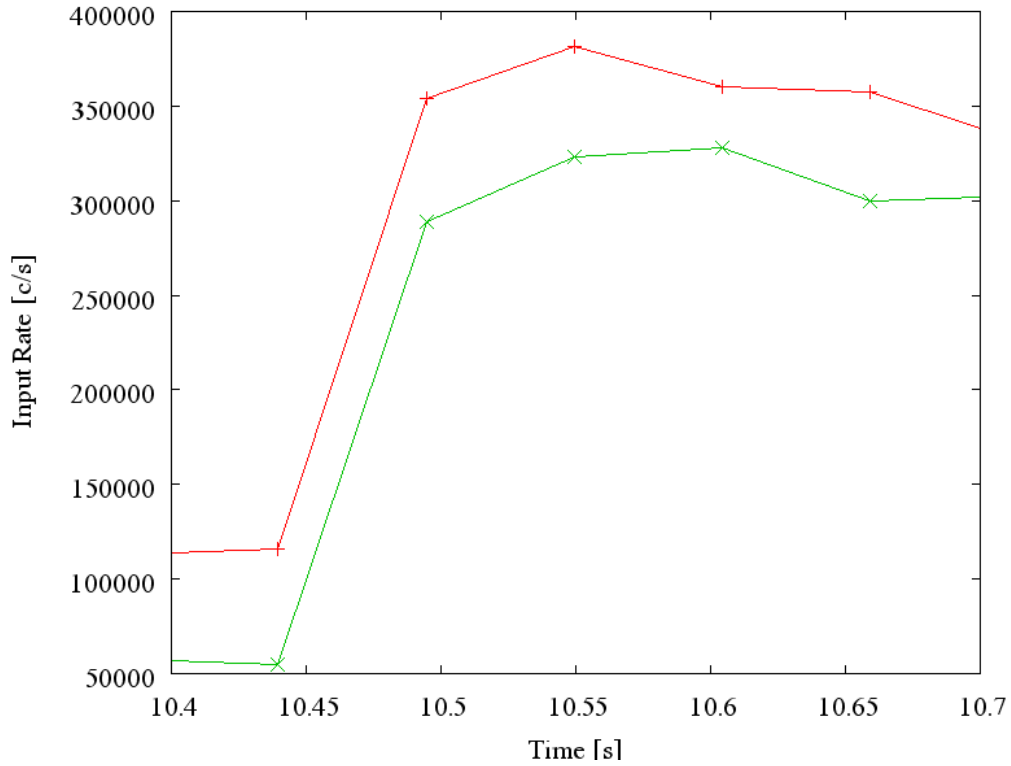


Figure 4.1: Part of the input-rate histogram showing arrival of the sample

nuclides it is obvious that the first spectrum is the most important in each sequence.

The input-rate histogram created by GSPEC can be used to accurately determine flight time. Figure 4.1 shows the important part of the input-rate histograms of two different samples. The input-rate starts to climb at 10.44 s and reaches a maximum 150 ms later at 10.59 s. Irradiation time was 10 s, so flight time was 300 ms.

Counting starts at the end of irradiation, so the first 600 ms of counting have to be dismissed. Considering that the flight-time for the sample going into the core and out to the detector is the same, irradiation time is kept exactly.

Sample Changer

As the detector almost touches the sample changer, exact positioning of the sample is very important to suppress geometry errors. The sample changer also has to meet detection position exactly because otherwise the sample

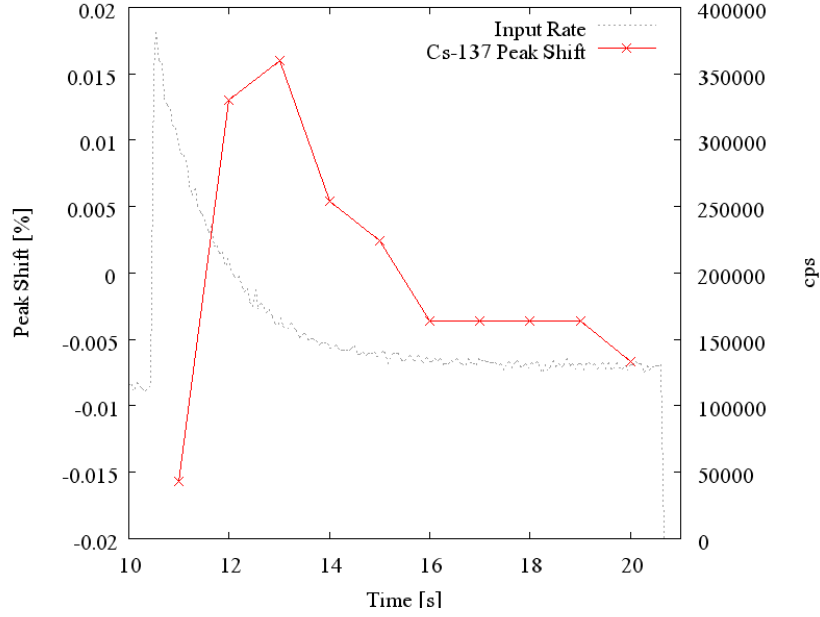


Figure 4.2: Peak shift of ^{137}Cs at variable counting rates

could be damaged while entering the transportation capsule.

To ensure exact and reproducible positioning, the SHOOT position is fixed with a little spring that has just enough strength to push the sample changer into the right position and provides enough resistance to keep the sample changer from moving during the impact of the returning sample. At the same time the motor of the sample changer is strong enough to move the sample changer out of the position to ensure flawless sample changes.

The positioning system was checked several hundred times and found working.

4.1.2 Detection System

Primary focus of the evaluation of the detection system was its behavior under rapidly varying input-count-rates. To this end, a simple experiment was devised. A ^{137}Cs source was placed next to the detector, providing a mean correction factor of 2.5. Additionally a sample of 0.04 g zirconium (half-life of $^{90\text{m}}\text{Zr}$ is 809 ms) was irradiated for 1 s and then measured during 10 spectra with 1 s counting time each. In this way, the peak of ^{137}Cs could be observed during input-rates rapidly changing from 400000 cps to 100000 cps.

With rapidly changing input-rates and therefore correction factors, energy and peak-area of the ^{137}Cs peak should stay stable. In figure 4.2, the peak-

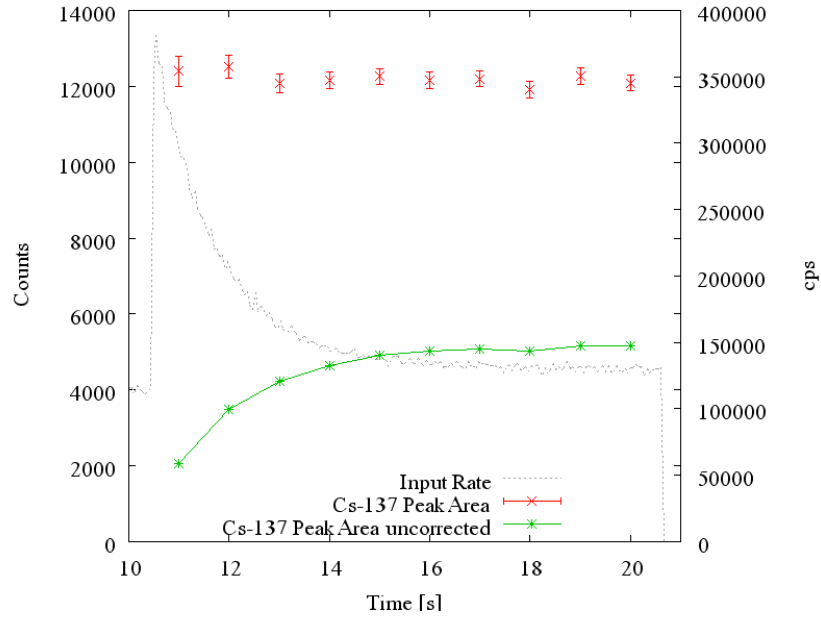


Figure 4.3: Corrected and uncorrected peak area of ^{137}Cs at variable counting rates

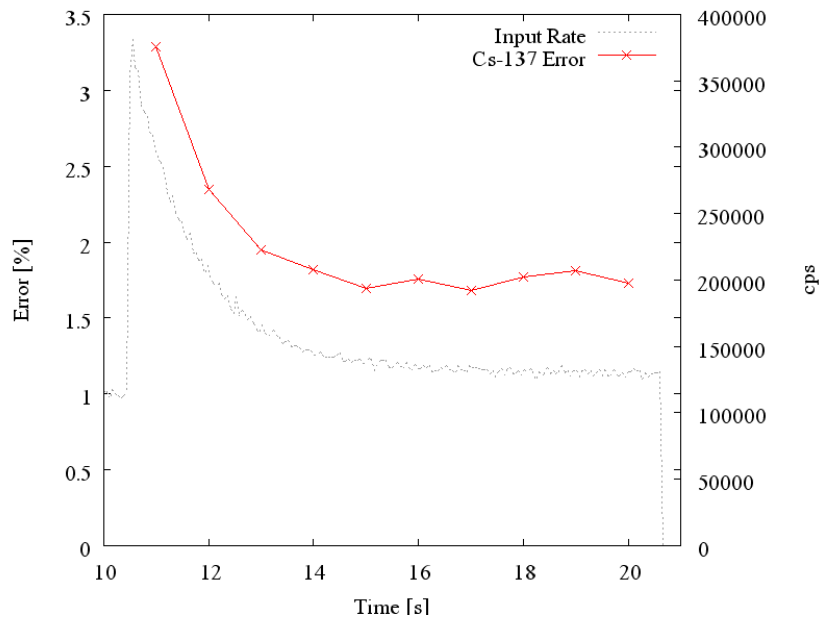


Figure 4.4: Counting error of ^{137}Cs peak at variable counting rates

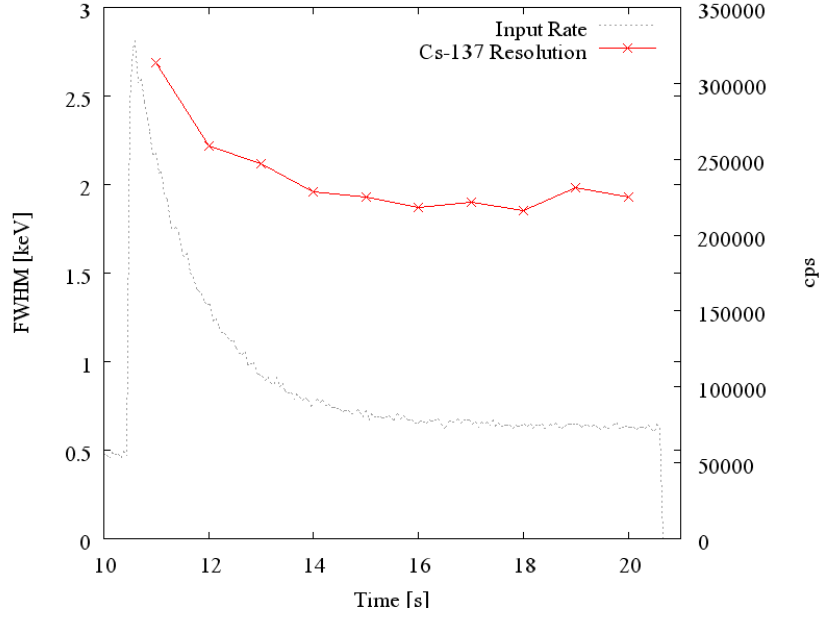


Figure 4.5: Peak resolution of ^{137}Cs at variable counting rates

shift of the peak can be found. The peak shifts at the most 0.015 % around the mean energy, which is more than reasonably stable.

Figure 4.3 shows both corrected and uncorrected peak-area during rapidly changing input-rates. While uncorrected values rise slowly, corrected values stay constant. Comparing figure 4.4, where the counting error of the corrected peak-areas is shown, the expected result of higher counting errors at higher input-rates or correction factors can be observed.

Figure 4.5 shows peak resolution of the ^{137}Cs peak. The resolution rises slightly above 2.5 keV at high input rates and drops back below 2 keV at input rates of 100000 cps.

To see how the corrections factors behave during the above measurement, GSPECs ability to produce histograms was used. Figure 4.6 shows three histograms taking during a repetition of the above experiment with the ^{137}Cs source much closer to the detector.

As can be observed, during the first second of the measurement, with the most rapid change of count-rate, the correction factors are distributed very widely with no discernable maximum. Only one second later, the distribution peaks at approximately 18. Another second later, the distribution is sharp and peaks at a correction factor of 10.

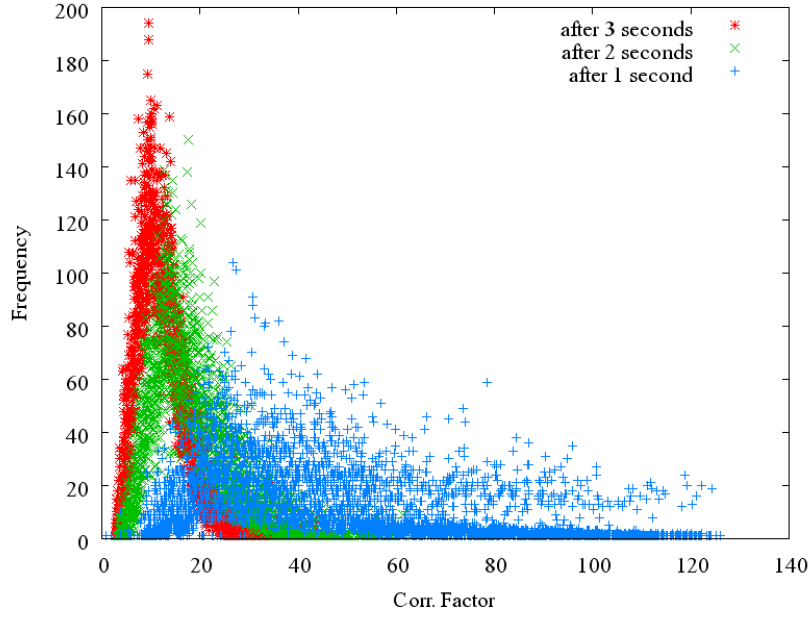


Figure 4.6: Histogram of correction factors

Time	Energy [keV]	mean con- centration	calculated error [%]	standard deviation [%]
13:30	133	0.8419	3.38	3.41
14:30	133	0.8538	4.61	4.74
13:30	2319	0.8006	2.07	2.27
14:30	2319	0.8016	2.22	1.82

Table 4.1: Flux stability measurements

4.1.3 Reactor

Neutron Flux Stability

To measure the neutron flux at the irradiation position a sample of 0,38 g zirconium was irradiated for 10 seconds. The short lived ^{90m}Zr ($\tau = 809.2$ ms) was measured at its two lines at 2319 keV and 133 keV in ten consecutive spectra with 1 second measurement time each. The calculated concentration of zirconium in the sample is directly proportional to the neutron flux.

The measurement was repeated ten times for better statistics and repeated after one hour to measure the stability of the flux. The results can be found in table 4.1.

The measurements at the 133 keV line have bigger errors because of the

much higher background at low energies. Using the virtually background free 2319 keV lines, the difference between the concentrations, and therefore the flux is less than one percent.

The measurements to obtain these results took about 10 minutes, so the errors of the mean values offer a value for the flux stability within a short period of time. Again, using the line at 2319 keV offers better statistics. The errors of the mean values for the two measurements again lie below 1 percent, so the short time stability of the neutron flux at the radiation position can safely be estimated with one percent.

During longer periods the neutron flux may change its absolute value because of recalibration of the reactor hardware and systems. It is therefore necessary to perform flux measurements regularly. The easiest way to do so is to measure a known sample again and compare the concentrations found. The ratio of the two concentrations can be used as a factor to adjust the saturation values in the main library.

4.1.4 Software

The two main programs, **GSPEC** and **NDANAEX** work flawlessly most of the time. **GSPEC** sometimes has problems finding the interface card, a bug that is being worked on. As of today that is the only problem encountered.

NDANAEX performs flawlessly all the time. Unfortunately it relies on the Nuclear Data program **PEAK** which seems to have problems with its memory management, so while processing large amounts of spectra, errors with **PEAK** occur.

GSPEC

GSPEC provides a user-friendly screen-interface if used manually, with self-explanatory text and options. When used with script-files, no unnecessary user input is needed, a feature that allows to call **GSPEC** several times from DOS batch-files.

Running on the current Pentium PC with 64 MB RAM, **GSPEC** can store up to 1000 pairs of corrected and uncorrected spectra in memory. The program itself could process count-rates of close to 2000000 cps, providing ample space for expansion of the detection system that ‘only’ works up to 1000000 cps.

There are some features that would make work with **GSPEC** even more user-friendly or convenient, mostly concerning the spectral view:

- Adding energy-calibration to the spectral view.

Sample	μg prepared		μg found	
	In	Se	In	Se
1	5.0	20.0	5.18 ± 0.71	18.5 ± 1.8
2	5.0	30.0	5.26 ± 0.73	33.5 ± 1.9
3	5.0	40.0	4.89 ± 0.15	42.2 ± 2.5
4	5.0	50.0	4.68 ± 0.03	46.4 ± 0.4

Table 4.2: Indium and Selenium values prepared and found in four samples

- Peak identification in the spectral view.

NDANAEX

The only problem occurring regularly with **NDANAEX** the already mentioned problem with **PEAK**. While **NDANAEX** is able to keep working through error messages from **PEAK**, sometimes **PEAK** causes a system crash, hanging up the PC. Nuclear Data never released source codes for their programs, so debugging **PEAK** is impossible. The best solution for this problem would probably be to program a new peak-fitting program, substituting **PEAK** altogether.

There are several parameters hard-coded into **NDANAEX**, like the width of the energy window while identifying peaks or output formats that are not accessible to the normal user. Providing support for an initialization-file that defines those parameters externally would provide more freedom for the average user.

Example output of **NDANAEX** can be found in listing 3.3, the most prominent feature though is **NDANAEX**' ability to separate otherwise coinciding lines using the algorithms explained in section 2.5. To evaluate this algorithm, four samples with different concentrations of Indium and Selenium were prepared and measured. The results can be found in table 4.2. The sample preparation was performed with unsuitable micropipettes by an inexperienced student, so the measured values might be closer to reality than the prepared values.

4.2 Sample data

For evaluation purposes several different standards have been measured. The results can be found in tables 4.3, 4.4, and 4.5.

IAEA-359, Cabbage			
Nuclide	found mg/kg	reference mg/kg	ratio
K	$34600 \pm 1.42\%$	$32500 \pm 2\%$	1.06
Mg	$2243 \pm 1.98\%$	$2160 \pm 2\%$	1.04
Mn	$29.1 \pm 1.47\%$	$31.9 \pm 2\%$	0.91
Ca	$18010 \pm 1.46\%$	18500	0.97
Na	$543 \pm 1.71\%$	580	0.94

Table 4.3: IAEA-359, Cabbage, reference values without error are non-certified values

NBS-1570, Spinach			
Nuclide	found mg/kg	reference mg/kg	ratio
Al	$659 \pm 0.66\%$	$820 \pm 10.98\%$	0.80
Br	$45.95 \pm 2.15\%$	$49.0 \pm 18\%$	0.94
Ca	$12600 \pm 1.64\%$	$13300 \pm 6.152\%$	0.95
Cl	$6220 \pm 1.29\%$	$6500 \pm 13.85\%$	0.96
K	$30300 \pm 2.40\%$	$35400 \pm 4.24\%$	0.86
Mg	$7630 \pm 1.78\%$	$8500 \pm 4.71\%$	0.90
Mn	$134 \pm 1.71\%$	$164 \pm 5.49\%$	0.82
Na	$12300 \pm 0.43\%$	$13900 \pm 7.19\%$	0.88
Sc	$0.135 \pm 3.75\%$	$0.165 \pm 6.67\%$	0.82
V	$1.18 \pm 4.58\%$	$1.20 \pm 14.17\%$	0.98

Table 4.4: NBS-1570, Spinach

SRM-1575a, Pine Needles			
Nuclide	found mg/kg	reference mg/kg	ratio
Al	$514 \pm 0,80\%$	$580 \pm 5.17\%$	0.89
Ca	$2800 \pm 3,83\%$	$2500 \pm 4.00\%$	1.12
Cl	$431 \pm 3,37\%$	$421 \pm 1.66\%$	1.02
K	$3590 \pm 9,26\%$	$4170 \pm 1.68\%$	0.86
Mg	$1096 \pm 22\%$	$1060 \pm 16.0\%$	1.03
Mn	$450 \pm 0,46\%$	$488 \pm 2.46\%$	0.92
Na	$100 \pm 3,82\%$	$63 \pm 1.59\%$	1.59
Rb	$17.8 \pm 12,4\%$	$16.5 \pm 5.45\%$	1.08

Table 4.5: SRM-1575a, Pine Needles

Chapter 5

Conclusion

The Neutron Activation Analyzer provides a very wide range of applications. Due to its very easy interface, simple sample preparation, high sample throughput, and plain-text output it can be used successfully by untrained personnel, offering easy and quick multi-element analysis. The unusually big sample size further reduces the danger of contamination and betters sample representation. Besides, apart from the obvious necessity of a neutron source, the whole setup is comparatively low-cost and easily maintained.

In view of these facts, the Neutron Activation Analyzer could be a very useful tool for developing countries that already have small nuclear research facilities, especially for analysis of soil-, food-, and mineral-samples.

At the same time, with its sophisticated scripting abilities, it can be a very powerful tool for the experienced experimenter. Separating single- and double escape lines from other nuclides, almost infinite expansion of the library due to its plain text nature, collection of several hundred spectra and multiple irradiation in a single measurement are only some of the expert features of the system.

List of Figures

2.1	Reaction chain for ^{23}Na and ^{37}Cl	7
2.2	Reaction chain for ^{35}Cl	8
2.3	Saturation activity	9
2.4	Detector chain	9
2.5	Gaussian and Semi-Gaussian shape	13
2.6	Diagram of a gated transistor reset preamplifier	13
2.7	Output signal of the gated transistor reset amplifier.	14
2.8	Block diagram of PLDF	15
2.9	Pulse response of pseudorectangular filter	16
2.10	Lookup tables of <i>DTC-CLC</i>	17
3.1	Front view of the measurement position.	24
3.2	The sample-rabbit separation process.	25
3.3	The sample changer.	26
3.4	Circuitry for the sample changer.	28
3.5	Circuitry for the central decoding unit.	29
3.6	Main screen of the program <i>GSPEC</i>	30
3.7	<i>GSPEC</i> Settings screen	31
3.8	<i>GSPEC</i> Acquire screen	32
3.9	<i>GSPEC</i> Test Interface screen	33
3.10	<i>GSPEC</i> Measurement screen	34
3.11	Inseparable peak of $^{116\text{m}}\text{In}$ and $^{77\text{m}}\text{Se}$	38
4.1	Input-rate histogram	48
4.2	Peak shift of ^{137}Cs	49
4.3	Peak Area of ^{137}Cs	50
4.4	Counting error of ^{137}Cs peak	50
4.5	Peak resolution of ^{137}Cs	51
4.6	Histogram of correction factors	52

List of Tables

2.1	Nuclides contained in 1 g of NaCl	6
2.2	Possible nuclear reactions for NaCl	7
2.3	Expected activities of the nuclides of 1 g NaCl	8
3.1	Data types for the <code>acquire</code> command	41
4.1	Flux stability measurements	52
4.2	Indium and Selenium values	54
4.3	IAEA-359, Cabbage	55
4.4	NBS-1570, Spinach	55
4.5	SRM-1575a, Pine Needles	55

Listings

3.1	<code>prepare.bat</code>	23
3.2	Example log-file	35
3.3	Example output of <code>NDANALYZ</code>	36
3.4	Example script file	42
3.5	Standard batch file	43
3.6	The library file <code>satur.txt</code> (abridged)	44

Index

- (n, α)-reaction, 6
- (n, γ)-reaction, 6
- (n, p)-reaction, 6
- activity, 7
 - saturation, **7**, 18
 - per mass, **18**, 45
- calibration
 - energy, 46
- dead-time, **14**
 - correction, 14, 16
- decay constant, 7, 18
- depletion layer, 11
- detectors, 8
- Fano-factor, 10
- GSPEC, 24, **29**
 - Acquire Mode, 32
 - Main Menu, 30
 - manual mode, 29
 - Measurement, 33
 - performance, 53
 - script mode, 29
 - Settings, 31
 - Test Interface, 33
- half life, 8
- header-file, 22, 46
- ionization, 10
- leakage current, 11
- library-file, 34, 35, **44**, 45, 46
- lithium drift, 11
- logging, 33
- Loss Free Counting, 16
- measurement, 39
 - fully automatic, 22, **41**
 - manual, 39
 - semi-automatic, 39
- multichannel analyzer, *see* GSPEC
- NDANAEX, 28, **37**
 - performance, 54
- NDANALYZ, 24, **34**
- NDCAL, 38
- NDCALS, 39
- NDHEADER, 38
- NDSUM, 38
- neutron capture, 6
- neutron flux, 6, 8
- nuclear reactions, 6
- overlapping peaks, **20**, 46
- PARS, 39
- pileup, **14**
 - correction, 16
- pneumatics, **24**
 - control, **27**, 33
 - performance, 47
- pre-amplifier, 8
- Preloaded Digital Filter, **14**, 17
- radiation, 10
- READPK, **39**, 44
- result.txt, 35, **36**

- sample changer
 - position
 - RELOAD, 26
- sample changer, **25**
 - control, **27**, 33
 - performance, 48
 - position
 - SHOOT, 25
 - UNLOAD, 26
- Schottky junction, 11
- script
 - commands, **40**, 40–41
 - file, 39, 53
- shaping time, 12
- thermal neutrons, 6
- tick, 40
- transportation time, 25, 47, **48**
- Virtual Pulse Generator, 16

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