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Chapter 4

A New Detection System

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4.1 Introduction

The modulation method (Chapter 3) proved to be an excellent experimental tool for the determination of piezoelectric moduli in a rather fast way, say within one hour.

However, applying the modulation method to study structural changes is limited because of the low speed in integrated intensity data collection. The necessity of good counting statistics becomes evident when effects of $\Delta H / H$ are about 0.1%. This means that the integrated intensity has to be sufficiently large. In case of the conventional zero-dimensional detector used so far, the maximum count-rate is limited to $1 \times 10^5$ cts s$^{-1}$ and therefore long measuring times or repetitive scans are needed. Taking a full data set of a piezoelectric crystal upon application of an electric field would require one year or more when a conventional X-ray source is used. Hence, fast and accurate data-collection is essential.

A significant decrease in data-collection time is obtained when a synchrotron source is used. An experiment spans, in general, a few weeks due to the large increase of the photon flux delivered by a synchrotron source. Here, the limiting factor is not the available photon flux but the maximum count-rate of the detector used. Therefore, in order to fully profit from the increased brilliance of the new X-ray sources, the development of a new detector system with a much higher maximum count-
rate and good detection quantum efficiencies for medium to high photon energies is required. In this case Si photodiodes, although also having a large maximum count rate\textsuperscript{11}, have too low detection efficiencies.

First, a scintillation detector (§4.2) as is used in the conventional modulation method will be discussed. The new detection system (§4.3) consists of the combination of a new developed detector (§4.3.1) in combination with a lock-in amplifier device (§4.3.2). Finally, the performance of the new detection system was tested in several piezoelectric experiments (§4.4. Chapter 5 and 6).

### 4.2 Scintillation Counter

The classic detection system used in X-ray diffraction crystallography is based on a scintillation counter\textsuperscript{2-4}. It consists of a scintillation crystal (Na\textsubscript{I} crystal containing 1\% Tl in solid solution) and a photomultiplier tube. When an X-ray photon falls onto the detector crystal it will be absorbed and several photons in the visible regime will be emitted. A part of these photons will be actually effective in the photomultiplier operation (=15\%) and free photoelectrons from the photocathode, which are successively multiplied in the photomultiplier. Finally, these photoelectrons are registered by the photomultiplier anode and the incorporated electronic system (i.e. pre-amplifier).

The maximum number of detected X-ray photons in a scintillation counter depends on the saturation limit of the photomultiplier used and electronic system and is in most cases 1·10\textsuperscript{5}-1·10\textsuperscript{6} photons s\textsuperscript{-1}.

### 4.3 New Detection System

A detector, originally used in IR-spectroscopy and having a larger dynamic range, was obtained and converted for X-ray photon detection. The combination of the detector with a lock-in amplifier decreased the measuring time significantly as will be discussed below.

#### 4.3.1 High purity Ge-detector

The purchased detector unit (403HS, Applied Detector Corporation) consists of a compact cylindrical liquid nitrogen cryostat which houses a cooled 50 mm\textsuperscript{2} high purity germanium crystal and preamplifier. The crystal thickness is 5 mm permitting high-detection efficiency for photon energies between 10 and 100 keV. The detector can be operated in either horizontal or inclined configurations and has a liquid nitrogen capacity for approximately 8 h and weighs 2.7 kg net.

The detector was originally developed for IR measurements in a current mode of operation in conjunction with a chopped signal source and synchronous lock-in signal detection. On request, the manufacturer replaced the standard sapphire IR window with an X-ray transparent beryllium window.

The preamplifier is a classic charge-feedback design, with a frequency response set by the feedback elements consisting of a 1 M\textOmega\ resistance in parallel with a 0.5 pF capacitor.
The germanium crystal

A cross section of the germanium crystal is shown in Figure 4-1a. The upper side of the crystal is smooth and faces the incoming X-ray photons. Opposite to this side a circular ring is etched away, so that the electric charges do not short-circuit the electrodes.

As the detector-diffractometer set-up was mounted onto a vertical translation table at the Materials Science beam-line (§3.4.2), a sampling scan of the detector crystal through the X-ray beam was performed. Figure 4-1b shows the measured curve with shoulders at either side of the main peak that arise as a result of the difference in charge-collection efficiency by various parts of the detector crystal. When X-ray photons are absorbed in the middle part (A) of the detector crystal, the charge-collection efficiency will be high due to the homogeneous electric field within the detector crystal. Hence, the detector response will correspondingly be large. However, absorption of X-ray photons at the edge (B) of the detector crystal will result in a small charge-collection efficiency, due to a diffuse curved electric field, and a correspondingly small detector response. X-ray photons absorption in the (C) region has a detector response between the other two responses, since this region is at the boundary of a homogeneous and diffuse electric field.

To avoid changes in integrated intensities due to a different charge-collection efficiency of the detector crystal during a scanning operation, a good alignment of the detector crystal, e.g. in the centre of the detector crystal, is necessary.

Principle of X-ray detection

Absorption of X-ray photons produce electron-hole pairs in the germanium crystal. They drift in the applied electric field (V=-300 V) to the electrodes and are converted to a voltage pulse by a charge-sensitive preamplifier. The energy required for creating an electron-hole pair is 3.0 eV. The number of electron-hole pairs is proportional to the energy of the absorbed X-ray photon.

The intrinsic efficiency, defined as the ratio of the number of pulses produced to the number of photons striking the detector, is close to 100% for a large energy range at the centre part (A, Fig. 4-1a) of the detector crystal.

Dynamic range

In the current integration mode of operation the voltage generated across the feedback resistance by the mean signal current limits the maximum measurable signal at an X-ray flux equivalent of \( \approx 2 \times 10^{10} \) photons s\(^{-1}\) for 10 keV X-rays and \( 2 \times 10^7 \) photons s\(^{-1}\) for 100 keV X-rays. The practical lower-frequency limit of operation is set by the DC drift of the preamplifier around a nominal value of \(-1\) V. This drift arises principally from the variation in the operating point of the input-junction field-effect transistor of the preamplifier and leakage current variations across the detector crystal itself. After an initial cool-down period of 1 h, the DC drift is \(<5\ \mu\text{V min}^{-1}\). To put this figure in perspective, note that a steady-state 60 keV X-ray flux of 300 photons s\(^{-1}\) generates a signal of 1 \(\mu\text{V}\). The measured root-mean-square signal noise for an averaging of 1 s is also 1 \(\mu\text{V}\) for the detector. If used to measure low X-ray fluxes (\(<1 \times 10^5\) photons s\(^{-1}\)) the detector may be operated in a
Figure 4-1: a: Cross section of detector's germanium crystal; b: Translation curve measured by the detector over full width of the internal Ge crystal. The shoulders are the result of different charge-collection efficiencies by various parts of the Ge crystal.
Figure 4-2: Spectrum of an $^{241}$Am source recorded with the germanium detector and a multi-channel analyser. The americium peak at 59.5 keV has FWHM of 3 keV.

**Photon-counting mode.** A good signal spectrum with a 3 keV FWHM noise figure is obtained from a 59.5 keV $^{241}$Am source when the output of the detector is post-amplified (Tennelec TC244, 2 $\mu$s shaping time, $10^3$ photons s$^{-1}$) and fed into a multi-channel analyser, see Figure 4-2. Note that pole compensation cannot be achieved with a standard nuclear spectroscopy amplifier, and this results in degradation of the spectrum at high-count rates. Using a simple single-channel discriminator and operating the detector in a single-channel counting mode eliminates the problems of drift associated with the current mode of signal measurements. Assuming measurements with X-ray energies above 20 keV, the only significant detector noise is that arising from X-ray background in the experimental hutch.

**Linearity test**

The linearity of the unit has been tested both in the low-flux region, where photon-counting mode is used, and in the high-flux region, where the current mode is used.

The linearity in the low count-rate region was tested at the RA source (Chapter 3) with a Mo target (17.45 keV). The incident flux was controlled by regulating the current setting of the generator. The AC output of the germanium detector was amplified by a factor of 1250, using an ORTEC 575A; the signal was subsequently treated by a single-channel analyser (ORTEC 550A) in order to eliminate electronic noise. The flux was measured both with the germanium detector and with a NaI scintillator (BEDE). The dead time of the NaI scintillator was calibrated beforehand, using the method of Chipman$^{[5]}$, and allows an exact determination of the true incoming number of photons.
Figure 4-3: Recorded flux as a function of incoming flux, using the photon-counting mode of the germanium detector. The solid curve shows the calculated response of the detector using a dead time of 1.9 μs.

Figure 4-3 shows the number of counts s⁻¹ recorded with the germanium detector as a function of true incoming number of photons s⁻¹ determined with the calibrated NaI scintillator.

The solid curve gives the calculated response of the detector using a non-paralysable dead time τ of 1.9 μs using \( N_r = \frac{N_i}{1 - N_i \tau} \) (Equation 4-1),

where \( N_r \) is the recorded photon flux, \( N_i \) the true incoming photon flux, and \( \tau \) the dead time of the detector. It should be pointed out that both Equation 4-1 and the dead time \( \tau \) depend on the time structure of the source used, and can be significantly different when used at the ESRF in 1/3 filling mode (Fig. 4-5a). It is seen that the unit can reliably be used up to a flux of \( 1.5 \times 10^5 \) photons s⁻¹ for the photon-counting mode, which is comparable with a standard NaI detector.

The linearity measurements for the high count-rate mode (current mode) were made at the Materials Science beam-line (Chapter 3). A monochromatic beam of 22 keV was used. The relatively low-energy was selected in order to be able to make a good comparison between the voltage given by the germanium detector and the flux measured with a calibrated Si photodiode. The Si diode was read out by a Keithly 486 picoammeter, and the germanium detector by a Keithly K2001.
multimeter. Figure 4-4a shows the recorded voltage given by the germanium detector as a function of the incoming number of photons. It is seen that the unit shows excellent linearity up to $1 \cdot 10^9$ photons s$^{-1}$ at 22 keV; above this value the preamplifier saturates. In Figure 4-4b, the voltage output of the detector in the low flux region is given. The figure shows that the current mode can be used reliably down to $3 \cdot 10^4$ photons s$^{-1}$ at 22 keV. Since both modes are used simultaneously and no switching between the two modes is needed, both output signals can be recorded during single scan and the region between $3 \cdot 10^5$ and $1.5 \cdot 10^5$ photons s$^{-1}$ can be used to scale the two modes together.

**Figure 4-4:** Output voltage of the germanium detector as a function of incoming photon flux (22 keV): a: The high flux range, b: An enlargement of the low flux region. The solid curve shows a fit of a straight line to the data points.
Time response

Since the detector is to be used in studies where the response of a crystal to an external perturbation is determined, its time response is an important characteristic besides maximum equivalent count-rate and dynamic range. The time response of the detector was tested using the time structure of the ESRF, which is given for 1/3 filling mode in Figure 4-5a. Figure 4-5b shows the response of the germanium detector recorded with a digital storage oscilloscope. The germanium detector is fast enough to see the super bunches, but not fast enough to separate the single bunches within each super bunch. The 1 μs response time is in agreement with the RC time constant of the preamplifier and the pulse duration in counting mode. It should be noted that in order to use the detector in current mode at the ESRF running in 1/3 or hybrid mode, low-pass filtering is used in order to average over the super bunches. This low-pass filter is chosen such as to average over the super bunches while being fast enough not to average over the perturbation applied to the sample.

4.3.2 Lock-in amplifier

In many fields of science a lock-in amplifier (LIA) is used to measure very small AC signals down to a few nV. Accurate measurements can be made even when the small signal is obscured by noise sources many thousands of times larger[6].

The LIA uses the technique known as phase-sensitive detection to single out the component of the signal at a specific reference frequency and phase. Noise signals at frequencies other than the reference frequency are rejected and do not affect the measurement[6].

Phase-sensitive detection

All lock-in measurements require a reference frequency. Typically an experiment is excited at a fixed frequency from a function generator or LIA, and the LIA detects the response from the experiment at the reference frequency. A schematic block diagram of the function of the DLIA is given in Figure 4-6a. A square-wave reference signal, as is shown in Figure 4-6b, is fed into a LIA. The LIA converts the square-wave to a sine-wave signal (i.e. representation of the signal in the frequency domain) with the same frequency as the external reference frequency. This sine-wave signal will be used as the new reference signal (Fig. 4-6c). An observed signal (Fig. 4-6d) with a certain frequency and phase will be amplified and multiplied by the reference signal internally. Mathematically speaking, sine waves of different frequencies are orthogonal, i.e. the average of the product of two sine waves is zero unless the frequencies are exactly the same. Therefore, in practice, a low-pass filter follows after the multiplier, and provides the averaging which removes the products of the reference with components at all other frequencies. This may yield a DC output signal proportional to the component of the signal whose frequency is exactly locked to the reference frequency.

Narrow band detection

The accuracy of the measurement depends heavily on the selected bandwidth of the LIA. A narrower bandwidth will remove noise sources very close to the reference frequency, whereas a
wider bandwidth allows these signals to pass. When high accuracy is of concern, a large time constant is preferred since the bandwidth of detection is inversely related to the (user selected) time constant ($\tau$) of the LIA’s low-pass filter.

Figure 4-5: a: Time structure of the ESRF in 1/3 filling mode, 1/3 of the ring is filled with 331 bunches, and 2/3 of the ring (661 bunches) is empty. b: Response of the germanium detector recorded with a storage oscilloscope.
Figure 4-6: a: Block diagram of the used LIA: Stanford Research Systems SR850, b: Arbitrary reference signal fed into a LIA, c: Reference signal (b) is converted to a sine-wave with corresponding frequency and a phase difference, d: A signal obtained from an experiment with its frequency and phase.
Measurement units

The digital lock-in amplifier (DLIA) of Stanford Research Systems USA (Model 850), as was used for this work, measures the first Fourier (sine) component of the square input signal at the reference frequency. The output signal of the DLIA is the root-mean-square of the amplitude of the first Fourier component and is expressed as $V_{\text{rms}}$.

Piezoelectric experiments

The basic principle of the DLIA in combination with a piezoelectric experiment is shown in Figure 4-7a. In a conventional non-perturbation single-crystal X-ray diffraction experiment one measures absolute intensities as is shown by $Q$ in the figure. Even in the modulation method (Chapter 3) absolute intensities are measured, although they correspond to different states of the applied electric field ($Q$ and $Q_+$.). In contrast, an intensity measurement with the DLIA, using the internal reference signal, results in a difference intensity ($R$) between the two states of the electric field. A certain time delay $\varphi$ can occur between the response signal and the reference signal, due to the experimental conditions such as slow electronics or too long signal-carrying cables.

The two values $x$ and $y$ are calculated by the DLIA using the internal $R$ and $\varphi$ values of the sine waves corresponding to the square-wave at the input. The mathematical relation between the possible outputs of the DLIA is shown in a polar plot as in Figure 4-7b.

Figure 4-7: Basic principle of lock-in detection. a: Difference in amplitude of two signals; b: As expressed in a polar plot.
4.4 Crystals in Electric Fields

The new developed detection system was tested with crystals in electric fields. Determination and temperature dependence of the piezoelectric constant $d_{33}$ of KTiOPO$_4$ was studied first. A second study, carried out with LiNbO$_3$ and DKDP, involved the changes in both Bragg angle and integrated intensity as measured by the counting method (modulation method) and the Ge-LIA system.

4.4.1 Samples

For the determination of the piezoelectric constant $d_{33}$ of KTiOPO$_4$ two samples were used. Both samples were cut along the crystallographic $c$-axis and have a plate-like shape, with sample (1) having the dimensions of 5x5x0.44 mm$^3$ and sample (2) of 4x4x0.33 mm$^3$. Furthermore, sample (1) was not polished, whereas sample (2) was polished to an optical quality. The samples were covered at the two largest sides with 1 µm thick Al electrodes and mounted in the same way as is discussed in §3.3.

For the other experiments, the same samples i.e. LiNbO$_3$ and KD$_2$PO$_4$ were used as were discussed in §3.3.

4.4.2 Results and discussion

**Piezoelectric constant $d_{33}$ of KTiOPO$_4$**

To obtain data for the determination of the $d_{33}$ of KTiOPO$_4$ (KTP) the three-step version of the modulation method (Chapter 3) was used in combination with the Ge-detector, set into the photon-counting mode. The (00l) reflections were measured for two different samples with an external electric field parallel to $c$-axis. The amplitude of the voltage was varied between 500 and 2000V and the frequency of the modulation was 33Hz.

Two series of measurements were carried out. For the first sample the measurements were carried out at the Materials Science beam-line (§3.4.2) using a wavelength of 0.564Å. The temperature dependence of the $d_{33}$ constant was measured on the crystal, on the RA source (§3.4.1) with Mo$_{Kα1}$ radiation with a high-resolution diffraction set-up. In both cases the sample was cooled by a nitrogen gas stream (Oxford Cryosystems, 600 Series). It is noted that by using high-energy synchrotron radiation very high resolution in reciprocal space could be obtained ($\sin \theta \lambda = 1.7\AA^{-1}$).

Figure 4-8 shows the shift $\Delta \theta$ as a function of $\tan \theta$ for crystal (1) at 100K, $3.4 \times 10^7$ Vm$^{-1}$ for the (00l) reflections with $l=20$, 22, 24...32, 36, measured with synchrotron radiation. The (0,0,34) reflection was influenced by multiple scattering and therefore not included in the data treatment. The shifts were determined by the program SHIFT (§3.5.1). The change in profile shape due to the applied electric field is very small, as was confirmed by the large correlation coefficient between the two profiles at the maximum overlap. All reflections and their Friedel equivalents have been measured between 10 and 30 times, with merged data presented in the figure. The solid line gives a linear fit to the data points$^{[1]}$. The slope of the curve gives a value of 15(2) $10^{-12}$ CN$^{-1}$ for the
piezoelectric constant $d_{33}$, which is between the values of $10.4 \cdot 10^{-12}$ CN$^{-1}$ and $25.8 \cdot 10^{-12}$ CN$^{-1}$ obtained by Chu et al.$^{[8]}$ and Sil’vestrova et al.$^{[9]}$ respectively. Both groups performed the measurements at room temperature using the direct piezoelectric effect but, unfortunately, give no indication of the precision of the results.

![Figure 4-8: Electric-field-induced peak shift for the (00l) reflections of KTiOPO$_4$ as function of tan $\theta$, where $l=20,22,24...32,36.$](image)

The peak shift ($\Delta \theta$) of the (0,0,36) reflection as a function of the applied electric field for crystal (2) at 100K is shown in Figure 4-9. From this the expected linear behaviour is evident. Table 4-1 lists a summary of the $d_{33}$ value obtained for different crystals and under different electric fields.

<table>
<thead>
<tr>
<th>Crystal #</th>
<th>Electric field $[\cdot 10^6 \text{ Vm}^{-1}]$</th>
<th>$d_{33}$ at 100 K $[\cdot 10^{-12} \text{ CN}^{-1}]$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3.4</td>
<td>14(2)</td>
</tr>
<tr>
<td>2</td>
<td>3.0</td>
<td>16(2)</td>
</tr>
<tr>
<td>2</td>
<td>4.5</td>
<td>16(2)</td>
</tr>
<tr>
<td>2</td>
<td>6.0</td>
<td>17(2)</td>
</tr>
</tbody>
</table>

Since the quoted literature values for the $d_{33}$ piezoelectric constant are obtained at room temperature, a study of the temperature dependence of the piezoelectric constant was performed. Piezoelectric tensor elements are, in principle, temperature dependent and show, in certain cases, large anomalies around phase transitions, e.g. $d_{36}$ of KH$_2$PO$_4$ (see Appendix B). The measurements were performed at the RA (MoK$\alpha$) on crystal (2) using an electric field of $6.7 \cdot 10^6$ Vm$^{-1}$. 

61
Figure 4-9: Induced peak shift for the (0,0,36) reflection for crystal (2) at 100 K as a function of applied electric field.

However, no anomaly in the $d_{33}$ value was observed as can be seen in Table 4-2, which is related to the fact that the transition at 150 K does not involve a change in symmetry. A least-squares fit of a line to the temperature data gave a temperature dependence of $d_{33}$ of $0.001(0.01) \cdot 10^{-12} \text{CN}^{-1}\text{K}^{-1}$.

Table 4-2: The $d_{33}$ value of KTiOPO$_4$ crystal (2) at various temperatures with an electric field of $4.5 \cdot 10^6 \text{Vm}^{-1}$ ($\sigma$ of $d_{33}$ not available).

<table>
<thead>
<tr>
<th>Temperature [K]</th>
<th>$d_{33}$ [$\cdot 10^{-12}$ CN$^{-1}$]</th>
<th>Temperature [K]</th>
<th>$d_{33}$ [$\cdot 10^{-12}$ CN$^{-1}$]</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>15</td>
<td>157</td>
<td>17</td>
</tr>
<tr>
<td>120</td>
<td>18</td>
<td>166</td>
<td>17</td>
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<td>140</td>
<td>16</td>
<td>180</td>
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</tr>
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<td>150</td>
<td>16</td>
<td>200</td>
<td>13</td>
</tr>
<tr>
<td>153</td>
<td>16</td>
<td>220</td>
<td>18</td>
</tr>
</tbody>
</table>

Note that the results obtained are believed to be sample independent, since the same value for the $d_{33}$ constant was obtained for two different crystals, measured at two different sources.

The Ge-LIA detection system

Since the Ge-detector gives a voltage output it can be readily used with a LIA to determine small changes in diffracted signal in perturbation experiments. The shifts of the diffraction peaks of a LiNbO$_3$ crystal (§3.3) in an external electric field were measured$^{[10,11]}$ using the Ge-detector and a
DLIA (Stanford Research Systems, SR model 850), referred as Ge-DLIA. An electric field of $3.8 \times 10^4$ Vm$^{-1}$ was applied in a two-step modulation with a frequency of 33 Hz. The measurements were performed at the RA.

Figure 4-10a shows a step scan of the (0,0,12) reflection where at each point of the scan the diffracted signal corresponding to both the positive and negative electric field has been recorded with a NaI scintillation detector (BEDE). The shift of the peak due the piezoelectric effect is clearly visible. The dashed curve in Figure 4-10b gives the difference between the positive and negative signal. The solid line in Figure 4-10b gives the change in the diffraction profile determined with the Ge-DLIA. At each step of the scan a single reading was taken, with integration time of the DLIA set to 300 ms. It can be seen that the two results are in good agreement.

Furthermore, Figure 4-10b also shows that with the DLIA differences down to $1 \times 10^4$ photons s$^{-1}$ at 17.45 keV can be detected using a time constant of 300 ms. This limit can be significantly reduced by increasing the integration time of the amplifier. This will, of course, increase the data-acquisition time per point and thus prolong the total scanning time per profile.

It should be noted that when using a LIA, only information about differences between the two states of the electric field is obtained (Fig. 4-10b), but no information about the individual peaks (Fig. 4-10a). To overcome this problem a Si-diode used in transmission can be mounted in front of the Ge-detector in order to determine the average profile. Since the absorption of the Si-diode is very small at energies above 25 keV, the signal of the Ge-detector is not influenced significantly.

Counting versus Ge-DLIA detection

Theoretically, changes in integrated intensities determined either by a counting method (as is used in the modulation method, Chapter 3) or by the LIA-Ge detection method should be identical.

The changes in integrated intensity measured by means of the two-step modulation method can be defined as

\[
\left( \frac{\Delta I}{I_0} \right)_{\text{counting}} = \frac{\sum I_{\text{+}} - \sum I_{\text{-}}}{\frac{1}{2} \sum (I_{\text{+}} + I_{\text{-}})},
\]

where \( i \) runs over all \( N \) data points in the profile and \( I_i \) is the integrated intensity corresponding to the rocking curve induced by a positive or negative electric field. After rearranging Equation 4-2 one obtains

\[
\left( \frac{\Delta I}{I_0} \right)_{\text{counting}} = \frac{\sum (I_{\text{+}} - I_{\text{-}})}{\frac{1}{2} \sum (I_{\text{+}} + I_{\text{-}})}.
\]
which also represents the behaviour of the Ge-DLIA system. The DLIA internally determines the difference of $I_1$ and $I_0$ (i.e. a hardware determination), which is then integrated by software. For the counting method software is used to calculate first the sum and then the difference. It should be noted that a single-point measurement for both methods should also give the same change in integrated intensity.
To validate this hypothesis experimental data taken at the High-Energy X-ray Scattering beam-line with the two-step modulation method, were evaluated for the (-4,10,-2) reflection of DKDP at one point only of the profile using an electric field of $1.3 \times 10^6$ Vm$^{-1}$ with a frequency of 30 Hz. The set-up consisted of two detection systems. The first detection system used the photon-counting mode of the Ge-detector whereas the second system used the current mode. A digital volt-meter (DVM, Keithley K2001) and the DLIA were joined together for the current mode detection. Here the DVM detects the average of the $I_+$ and $I_-$ signal, giving $I_0$.

The values obtained for the changes in integrated intensity measured by the DLIA using a small time constant $\tau \leq 300$ ms do not correspond to the values measured by the counting method. This can be understood as follows. Using a small time constant implies that the bandwidth of detection is large and a limited sampling of the input-signal by the DLIA occurs. To illustrate this, a time constant of 33.33 ms would sample only one period of the electric field, hence giving for each measurement a different value for the change in integrated intensity. Using, however, a larger time constant of, for example, 333.33 ms would result in a sampling of 10 periods and narrow bandwidth detection.

So, taking a time constant of 10 s for the DLIA showed that the obtained values of changes in integrated intensities agreed for both methods. The $\sigma(\Delta I/I)$ ratio for the DLIA values are about 1%. Using time constants of 3 and 1 s showed that the ratio increased from $=1.27$ to $=1.58\%$, respectively. Decoupling the DVM and the RC filter (belonging to the Ge-detector) from the current mode detection system, showed that the ratios are about 0.6% for a time constant of 10 s.

Since the Ge-DLIA detection method was developed for fast data collection, a time constant of 10 s is obviously inappropriate. Therefore, taking the results discussed above into account, a time constant of 300 ms should be a good compromise for both good counting statistics and fast data collection.

### 4.5 Conclusion

The tested 403HS germanium detector has shown to be linear both in a low-flux counting mode ($<1$ to $1 \times 10^5$ photons s$^{-1}$) and in a high-flux current mode operation (equivalent count-rate up to $1 \times 10^9$ photons s$^{-1}$). The dead time in photon-counting mode is 1.9 $\mu$s, which is comparable with a standard NaI scintillator detector. There is sufficient overlap between the photon-counting and current mode for scaling the two ranges together. This is especially true when the current mode is used in conjunction with a chopped signal and synchronous lock-in detection, in which case signals below $1 \times 10^3$ photons s$^{-1}$ can be measured for signal integration times of 300 ms. The detector has a time response in the order of 1 $\mu$s, making it a suitable detector for perturbation measurements in general and for crystals in electric fields in particular.

Ideally, the detector should incorporate two preamplifiers. An electrometer optimised for low drift and low noise for current mode operation with slowly varying signals, and a charge preamplifier
suitable for post amplification by a high-rate nuclear-spectroscopy amplifier incorporating base-line correction for rapidly varying signals. This last option should be used either in photon-counting mode for weak X-ray fluxes, or in current mode at high fluxes using the lock-in amplifier technique to compensate for DC drift. The present detector provides a compromise solution between these two ideals, offering a low-price system of wide dynamic range.

References