

## Characterization of emission spectra of an Isovolt 3003 x-ray tube by using a fast readout charge-coupled device

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### Abstract

**Introduction:** In the framework of this thesis, experiments were done to make precise measurements of X-rays using Fast Readout Charge-Coupled Device (pnCCD).

**Aim:** This study has been done to characterize the radiation emitted by the primary & secondary X-ray source of the apparatus. As a part of this experiment, it is necessary to measure the emission spectra of the X-ray tube itself and then the emission spectra of the manganese fluorescence X-rays excited by that radiation, eg, in manganese dioxide ( $MnO_2$ ).

**Method:** Two cases were considered here during the experiments.

Case 1: Measurement of X-rays emitted from the X-ray tube itself. In this case highly accelerated electrons hit the anode material and resultant X-rays are detected by the detector.

Case 2: Measurement of fluorescence X-rays from the secondary target ( $MnO_2$ ). In this case, X-rays are emitted by the secondary source where the characteristic fluorescence radiation of manganese (Mn) is excited by means of X-ray tube.

**Result:** In the 1<sup>st</sup> case many chemical elements have been observed in the spectrum. These are the elements of X-ray tube and its surrounding materials. By using the energy calibration, the elements have been clearly identified.

In the 2<sup>nd</sup> case, a clean Mn spectrum has been measured. The secondary target is surrounded by the aluminum foils that shield the X-rays from other elements, thus decreasing the background.

**Conclusion:** In all the cases, the degree of illumination of the pnCCD is kept below 2% not to distort the intensity determination by applying aluminum filters of suitable thickness. The intensity of the K X-rays emitted from the secondary target is about 0.06% compared to the total intensity of the direct emission spectrum of the X-ray tube for 25 keV.

**Keywords:** Institut für Kernphysik, Forschungszentrum - Germany, Kepton window, pnCCD, FITOS, Bremsstrahlung

### Introduction & motivation

Since the discovery, X-ray techniques are used as great analytical tools worldwide for many applications, like material characterization and medical imaging and are still used for basic research. So X-ray

emission spectra, e.g., reveal details of the atomic structure like the effect of chemical compounds on the energy levels even for inner electron shells [1].

Each element's emission spectrum is unique, because of a unique set of energy levels. Therefore each element emits a pattern of X-ray characteristics of the elements, so called "characteristic X-rays" and X-ray fluorescence method can be used to identify the elements in a material of unknown composition.

Using an X-ray tube, a spectrum is observed characteristic for the material that is used as the target in the tube and a continuous spectrum originating from Bremsstrahlung upon which is superimposed to the line spectrum.

This work is done within studies using X-rays to excite atoms from secondary targets in order to study their atomic shell structure by means of an ultimate resolution crystal spectrometer [2]. The radiation is produced by a commercial X-ray tube available at the Institut für Kernphysik at Forschungszentrum, (Institute of Nuclear Physics, Research center) Jülich, Germany.

**Experimental method**

This experiment has been divided in two phases.

- (i) The first phase focuses on the study and characterization of X-ray emission spectra from the X-ray tube itself (for different accelerating voltage settings).
- (ii) The second part involves the measurement of fluorescence X-rays emitted from a secondary target and possible background contributions from scattered radiation (in this case MnO<sub>2</sub>) which was excited by the radiation studied in phase (i). The same high-voltage settings from 5-60 keV were used in both phases.

**Setup to measure the X-ray tube emission spectra**

The experimental set up is shown below. The X-ray tube emits in line with the detector main axis. The distance between the detector and the X-ray tube was measured to (150.5 ±1) cm. Two plastic collimators (2 cm thick and opening 30 mm in diameter) are placed inside a coupling tube. The first collimator is placed after the Kapton window and half way between X-ray tube and detector to minimize background from scattered radiation. The tube is evacuated to reduce the absorption losses.

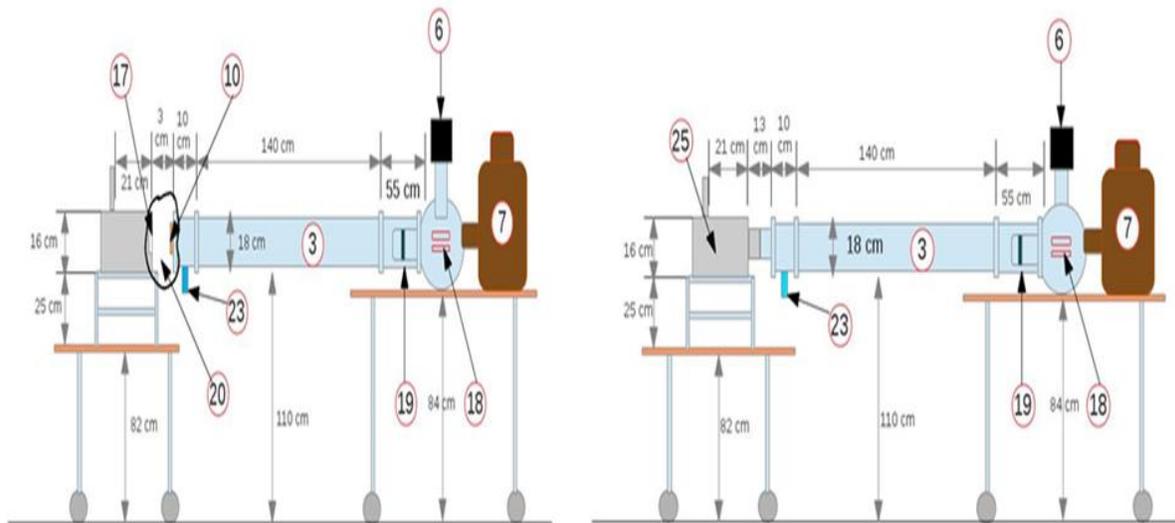
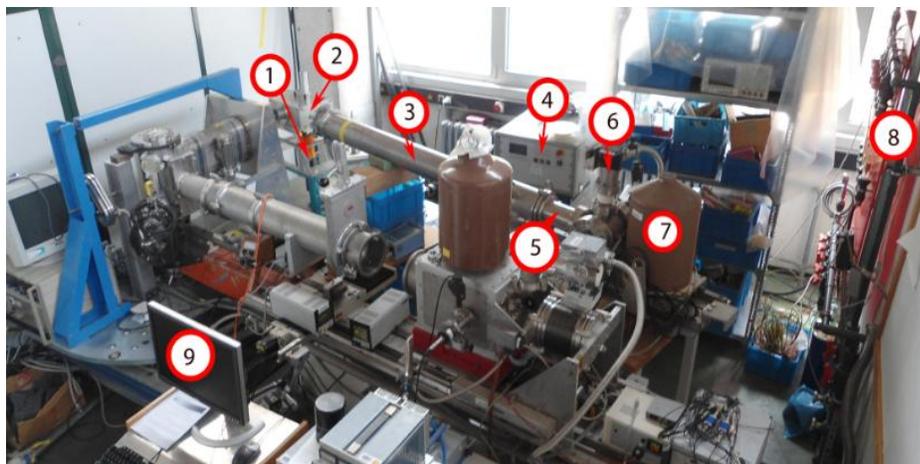


Figure 1: phase1 & Phase 2 (Sectional view).



**Figure 2: Over all view of the X-ray tube operation room** 1) Housing of the X-ray tube, 2) position of the Kapton window, 3) vacuum tube, 4) high-voltage generator, 5) position of Be window, 6) detector turbo molecular pump, 7) detector liquid nitrogen dewar, 8) X-ray tube water cooling distributor, 9) data acquisition computer.

### pnCCD setup

The following figure shows the connection of the pnCCD detector to the vacuum tube. The X-rays coming from the X-ray tube via the source is directed to the pnCCD detector.

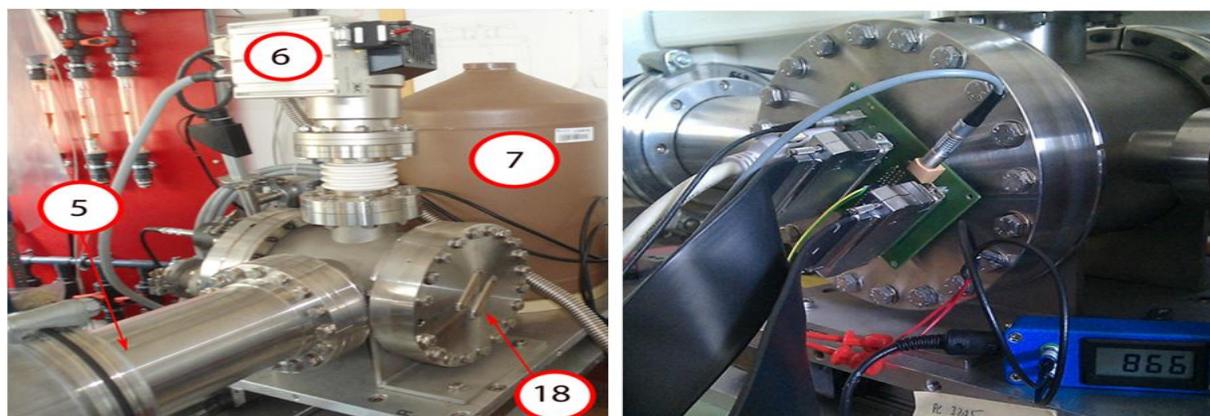
### Setup to measure the emission spectra of the $\text{MnO}_2$ target

Here, as source acts a  $\text{MnO}_2$  target that is surrounded by pure Aluminum so that only the manganese X-rays can be seen in the spectrum.

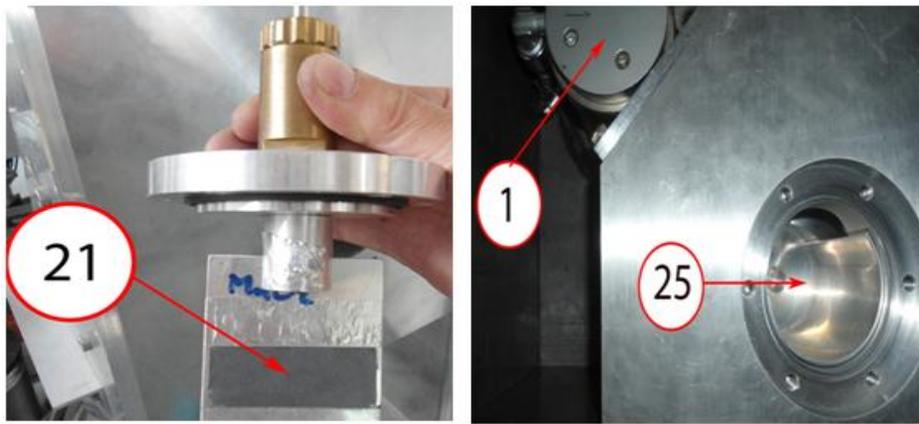
### Data acquisition

There are three main programs that work together with specific hardware resources

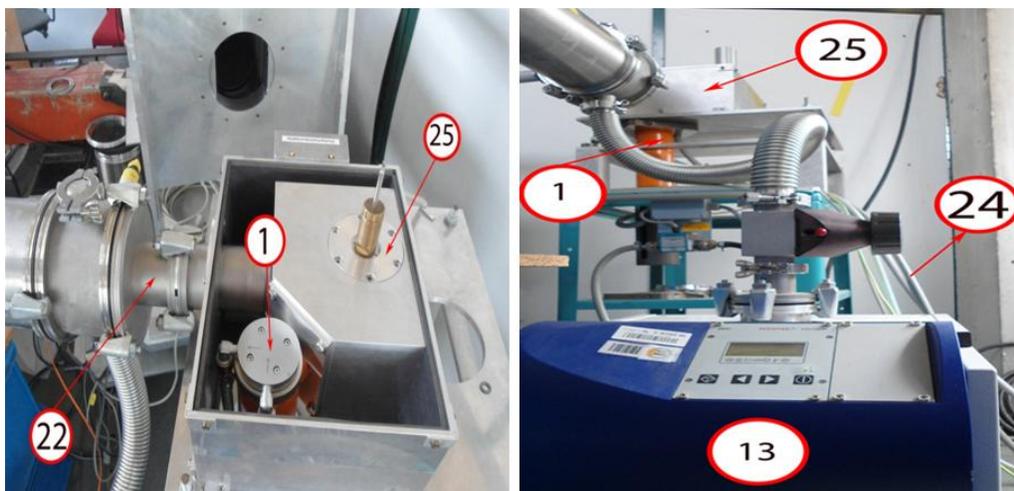
for the data readout and acquisition from the pnCCD chip. Of them Power Control program is used to power up the outer electronics crate serving the detector. CCD Control program is used for detector control. This is the main software, which communicates with the PnCCD. pnDataDisplay program is to generate an online spectrum and save it as txt files that are used by “gnu plot” for further analysis. The final txt files consisting of the digitized data that are used for plotting the spectra.



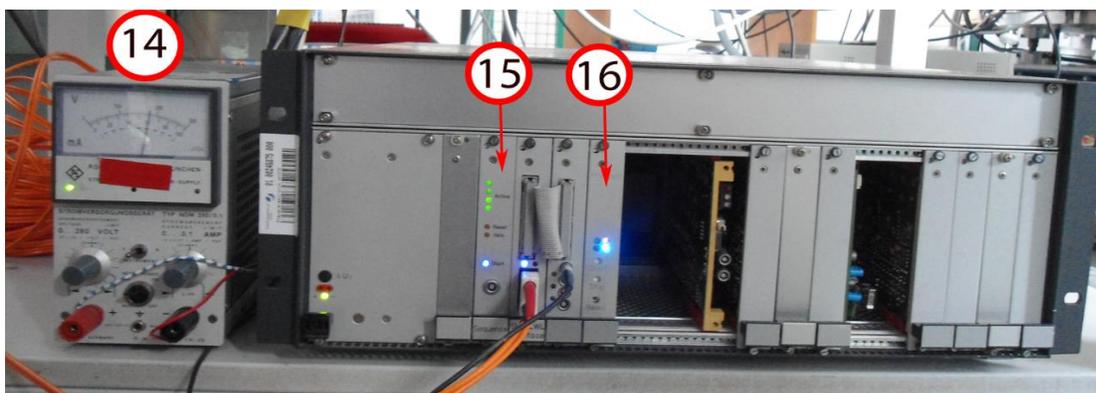
**Figure 3: Position of the pnCCD detector.** 5) Position of the 30  $\mu\text{m}$  Be window, 6) detector turbo molecular pump, 7) liquid nitrogen dewar.



**Figure 4: Top view of the X-ray tube with source position.** 1) X- ray tube, 25) Position of MnO<sub>2</sub> target, 21) Source MnO<sub>2</sub>.



**Figure 5: General view of source and coupling tube to detector.** 1) X-ray tube, 25) position of the secondary source, 22) coupling tube between X-ray tube and detector.



**Figure 6: Outer electronics crate.** 14) Power supply for the detector bias voltage, 15) Sequencer, 16) PROFIBUS (inter connection standard for backplane modules).

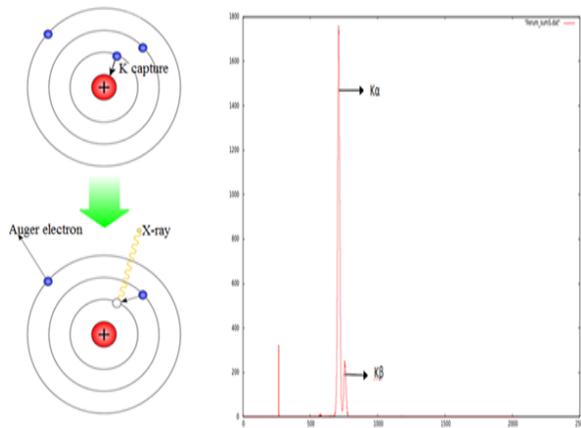
Afterwards the user can see a window on a screen plotting online data. First the noise peak will appear, after 10 seconds put the

source in front of the detector. Changes can be seen immediately on a screen.

**Calibration and analysis**

**Energy Calibration using a radioactive source**

To identify the corresponding energies of each peak, it is needed to know the energy corresponding to each channel. In our case, the calibration of the detector is done by using a radioactive Fe-55 source that emits Mn-55 X-rays after electron capture:  $e^- + {}^{55}\text{Fe} \rightarrow {}^{55}\text{Mn} + \nu$ . Fe-55 decays via electron capture to manganese Mn-55, shown below [3].



**Figure 7: Fe-55 electron capture process and subsequent X-ray emission.**

The position of the noise peak, set to ADC channel 250 by software, corresponds to X-ray energy zero. Most of the noise peak is already cut by the data acquisition software to keep the amount of stored data small.

The first peak in the spectrum starting from the left is the remaining tail of the noise peak. This offset needs to be subtracted from each channel for a proper energy calibration. The most prominent peak represents the  $K\alpha$  line of Mn located at channel 706. So if the offset is subtracted, the real value of the channel becomes 456. From the literature, it is known that  $K\alpha$  energy for Mn is 5892 eV (5.892 keV). Consequently the offset corrected spectrum yields that the energy interval per channel is  $5892 \text{ eV}/456 \text{ ch} = 12.921 \text{ eV/ch}$  (ch=ADC channel). This is called the energy calibration or energy gain

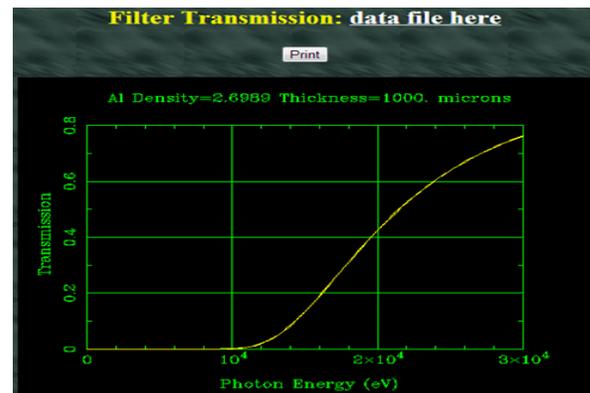
factor and is assumed to be constant for the whole spectrum.

Based on the energy gain factor, the energy of each peak can be calculated. If these values are compared to the photon energies and intensities of the X-ray data booklet table, materials excited can easily be identified.

**Degree of the detector illumination**

Any over illumination of the detector leads to an underestimation of the count rate, because the charge collected in one detector pixel can't be identified as a single X-ray event. An illumination degree of 2% or less is assumed to be a safe limit. To ensure the safe limit of the illumination degree (less than 2% or equal) it is needed to use filter.

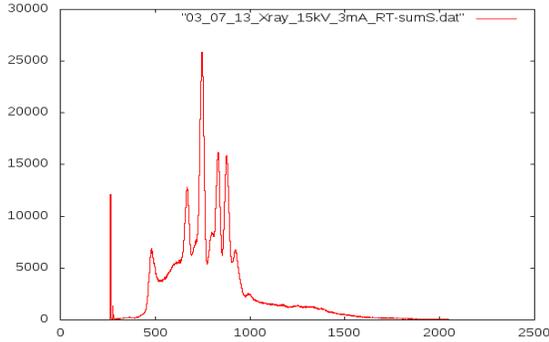
To get the required thickness of the aluminum filters to be applied in our case, we can use a calculator provided by NIST [4]. If the thickness of the Aluminum filter is 50 micron, more than 50% photons penetrate for energy lower than 8 keV leading to over illumination of the detector. The optimal value of thickness of the aluminum filter has been found to be 1000 micron as around 70 per cent photons are allowed to reach the detector for the highest possible energy in case of 25 keV.



**Figure 8: Filter transmission at filter thickness 1000.**

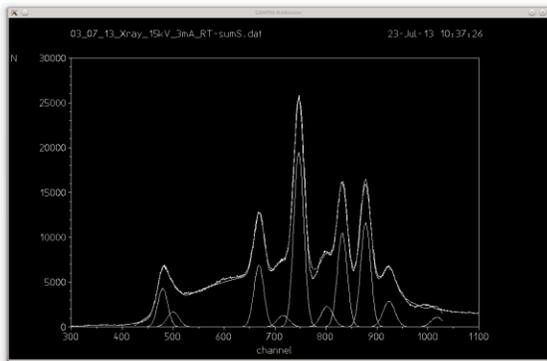
**Analysis procedure**

Let's consider the final spectrum obtained at 15 kV in order to describe the analysis procedure.



**Figure 9: Spectrum obtained at 15 kV with 3 mA.**

Because of the partly very small differences between adjacent peaks, some peaks are even overlapping, that means they are not clearly visible. By using the fitting technique the existence of such peaks in the spectrum can be better analyzed. The fit delivers results as the final positions for each of the peak of the spectrum, their width, intensities and corresponding errors. Fitting of the spectral lines has been done by the computer program so called "FITOS" [5]. The best fit to the spectrum obtained for 15 kV is shown below.



**Figure 10: Screen shot displaying the fit to the spectrum for 15 keV high voltages.**

Because of the very narrow differences between two adjacent peaks, some peaks could be hidden, meaning that they are not clearly visible. But under the fitting technique the adjacent peaks in the spectrum can be clearly visualized.

**Results**

**Spectra emitted by the X-ray tube**

The table shown below presents the literature value of the elements found from our experiment. So we can match our experimental value with this literature value to be sure of the identity of the elements (Table 1).

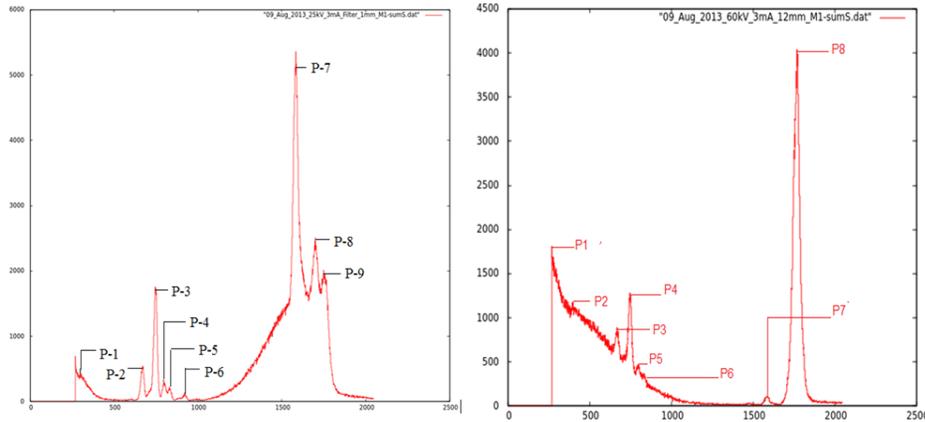
**Table 2: X-ray energies are taken from reference [6].**

Elements	Atomic number (Z)	Energy /KV	Transition
Ar	18	2.957	K $\alpha$
		3.192	K $\beta$
Cr	24	5.414	K $\alpha$
		5.946	K $\beta$
Fe	26	6.403	K $\alpha$
		7.057	K $\beta$
Co	27	6.930	K $\alpha$
		7.649	K $\beta$
Ni	28	7.47	K $\alpha$
		8.263	K $\beta$
Cu	29	8.042	K $\alpha$
		8.904	K $\beta$
Zn	30	8.638	K $\alpha$
		9.571	K $\beta$
Mo	42	17.47	K $\alpha$
		19.61	K $\beta$

At 25 kV excitation energy, a lot of elements have been excited leading to the corresponding peaks. Using energy calibration factor we can find out the corresponding energy of each peak. The most significant difference appears in peak 7 as molybdenum (Mo) X-rays from anode material becomes visible.

**Table 1: Measurement parameters in phase 1.**

<b>Applied Voltage (kV)</b>	5	10	15	20	25	30	35	40	45	50	55	60
<b>Current (mA)</b>	3	3	3	3	3	3	3	3	3	3	3	3
<b>Thickness of Al Filter (mm)</b>	-	0.1	2	0.5	1	2	3	4	6	8	10	12

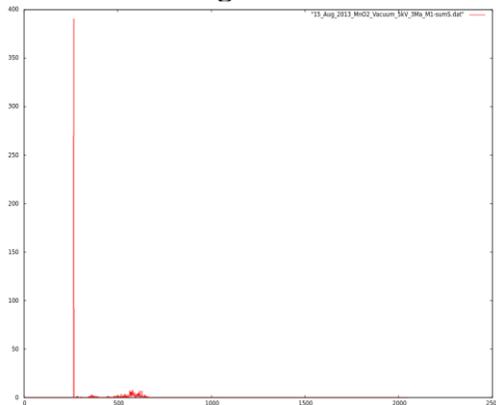


**Figure 11: X-ray spectrum measured at 25 kV (Left) high voltages: P1 (unidentified), P2 (Cr K $\alpha$ ), P3 (Fe K $\alpha$ ), P4 (Fe K $\beta$ ), P5 (Fe K $\beta$ ), P6 (Zn K $\alpha$ ), P7 (Mo K $\alpha$ ), P8 (Mo K $\beta$  backscattering), P9 (Mo K $\beta$ ). X-ray spectrum measured at 60 kV (Right) high voltages: P1 (Unidentified), P2 (P K $\alpha$ ), P3 (Cr K $\alpha$ ), P4 (Fe K $\alpha$ ), P5 (Fe K $\beta$ ), P6 (Ni K $\alpha$ ), P7 (Mo K $\alpha$ ), P8 (Mo K $\beta$ ).**

At the excitation voltage of 60 kV, overflow of data has been observed, meaning we can't see any peak over 20 keV X-ray energy because at this stage the detector can't detect the X-rays because it exceeds the maximum capacity of our experimental detector. The spectrum ends with the 20 keV peaks because of the range of ADC.

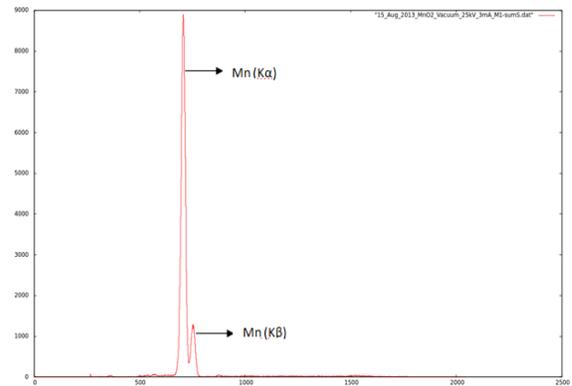
few events representing Al but we cannot observe any Mn X-ray spectrum from the figure shown below because the ionization energy of manganese is 8 keV, so 5 kV HV is not enough to ionize the manganese.

**Spectra emitted by the secondary fluorescence target MnO<sub>2</sub>**



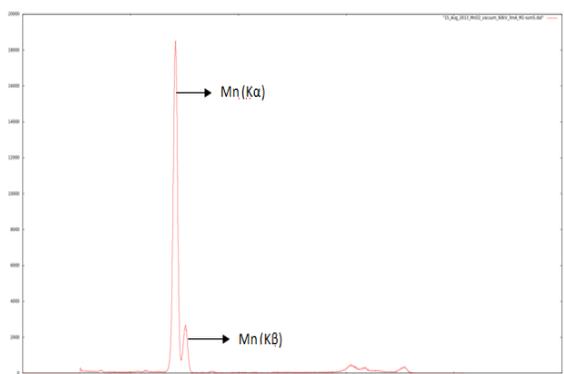
**Figure 12: X-ray spectrum measured at 5 kV high voltages.**

The spectrum was acquired at 5 kV high voltage and at this voltage level we get a



**Figure 13: X-ray spectrum measured at 25 kV high voltages.**

In case of 25 kV, Mo just begins to be ionized as its ionization energy is 19.87 keV but the energy is not high enough to present a clear spectrum. But the most significant contribution from Mo (shown below) appears at 60 kV.



**Figure 14: X-ray spectrum measured at 60 kV high voltages.**

### Summary & discussion

In phase 1, X-rays are measured as emitted directly from the X-ray tube itself. Initially high speed electrons hit Mo anode producing mainly a continuous Bremsstrahlung spectrum. This radiation produces fluorescence X-rays of different elements in the surrounding materials. The characteristic X-rays of the surrounding materials, mostly Cr and Fe are identified and together with a strong contribution from Bremsstrahlung.

From the excitation of the secondary target ( $\text{MnO}_2$ ) in phase 2 almost only Mn X-rays has been observed. There is no contamination from fluorescence of other elements due to a pure Al surrounding. There is a negligible contribution of flat background radiation originating from scattered X-rays. The Al fluorescence of 1.45 keV is suppressed by absorption and the low fluorescence yield.

### Acknowledgements

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### References

- [1] Anagnostopoulos, Sharon, Gotta, and Deutsch, *K $\alpha$  and K $\beta$  X-ray emission spectra of metallic scandium*, Phys. Rev. A 60 (1999) 2018
- [2] B.H Bransden & C.J Joachain, *Physics of atoms & molecules-2<sup>nd</sup> Ed*, Pearson Education Ltd, Essex, England 2003, pp.39-41
- [3] K.Heyde, *Basic ideas & concepts in Nuclear physics-3<sup>rd</sup> Ed*, Bristol & Phila Delphia, 2004, p.293
- [4] X-ray transmission factors: [http://henke.lbl.gov/opticalconstants/filter\\_s2.html](http://henke.lbl.gov/opticalconstants/filter_s2.html).
- [5] *Program FITOS*, D. Gotta et al., priv. com
- [6] *X-ray energies*: <http://www.csrii.iit.edu/periodic-table.html>.