XRF Lunar Spectrometer

XRF : Executive Summary Report

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This document is the Executive Summary Report for

ESA Contract No. 4000130471/20/NL/BJ/va
"Preparation of enabling space technologies and building blocks"
"Lunar XRF Spectrometer"

The ESR is a mandatory deliverable, due at the end of the contract.

1. Introduction

X-ray Fluorescence (XRF) is a spectroscopic method that allows detection and measurements of the amount of any atoms heavier than Ne. XRF spectrometers are used in wide range of application and are paramount instruments for planetary science since the beginning of space exploration. Improvements of detectors and electronics allowed to improve capabilities of such planetary instruments. Up to now, all the XRF space instruments have used radioisotopes sources as excitation sources. The only exception is the very last PIXL instrument sent on Mars by NASA on-board Perseverance, which uses an X-ray tube.

Because in space the weight and power consumption are critical parameters, we proposed to use a lightweight Pyroelectric X-ray Generator (PXG) as X-ray excitation source for XRF process. The XRF photons are detected using a Silicon Drift Detector (SDD), custom electronic readout, special algorithm and renormalization process.

The current development using pyroelectric X-ray generator as an excitation source has reached a Technology Readiness Level between 6 (full-scale model demonstration in relevant environment) and 7 (Model demonstrating the element performance for the operational environment).

2. Physical principle:

An X-Ray source irradiates the sample and X-Ray fluorescence occurs; a detector counts incoming X-Ray photons and measures their energies. The X-Ray fluorescence spectrum is recorded and analysed. X-Ray Fluorescence is the emission of characteristic "secondary" (or fluorescent) X-rays from a material that has been excited by X-rays or gamma rays. When materials are exposed to short-wavelength X-rays or to gamma rays, ionization of their component atoms may take place. Ionization consists of the ejection of one or more electrons from the atom, and may occur if the atom is exposed to radiation with an energy greater than its ionization energy. X-rays and gamma rays can be energetic enough to expel tightly held electrons from the inner orbitals of the atom. The removal of an electron in this way makes the electronic structure of the atom unstable, and electrons in higher orbitals "fall" into the lower orbital to fill the hole left behind. In falling, energy is released in the form of a photon, the energy of which is equal to the energy difference of the two orbitals involved. Thus, the material emits radiation, which has energy characteristic of the atoms present

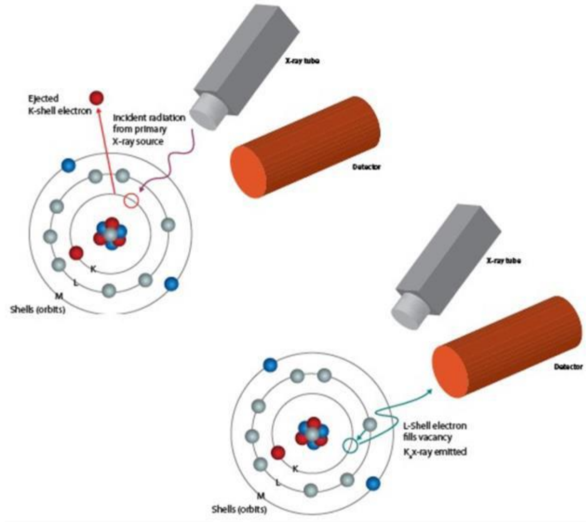


Figure 1 : XRF principle

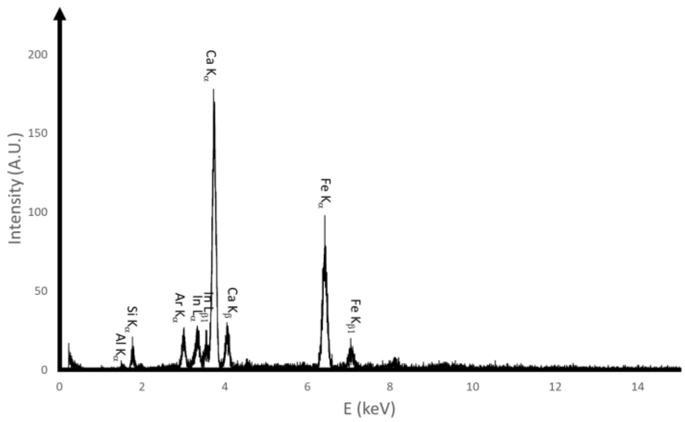


Figure 2 : Measured XRF spectrum

Figure 2 shows a typical spectrum measured with our instrument on anorthosite (Moon soil simulant) sample at atmospheric pressure. An Indium wire is present in the field of view of the instrument for spectrum renormalization. XRF energies and corresponding atoms are identified.

The general principle of the instrument is illustrated on Figure 3

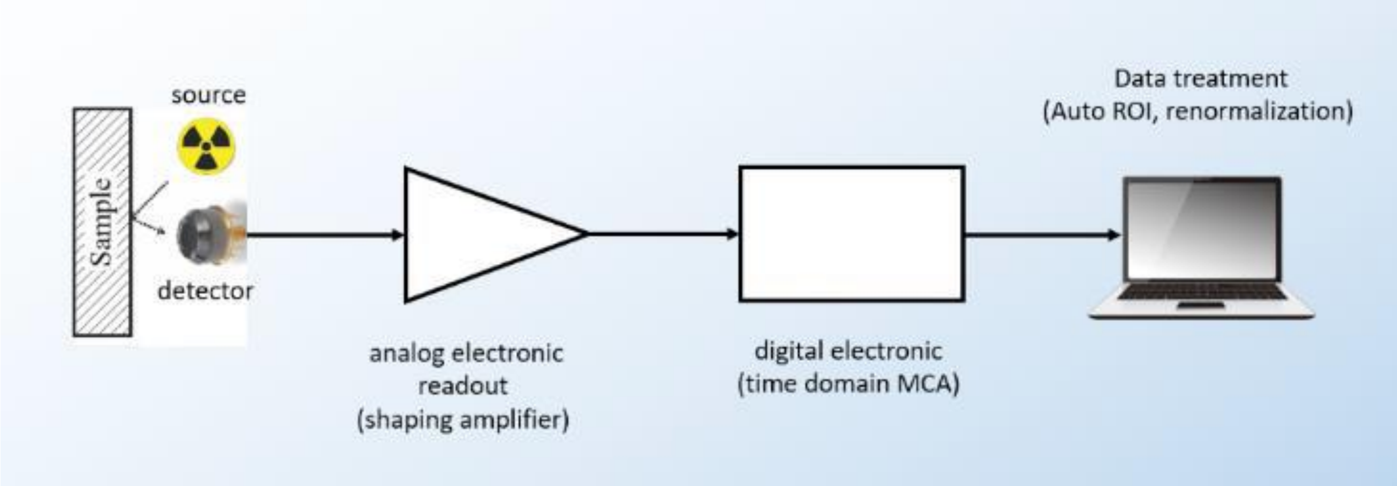


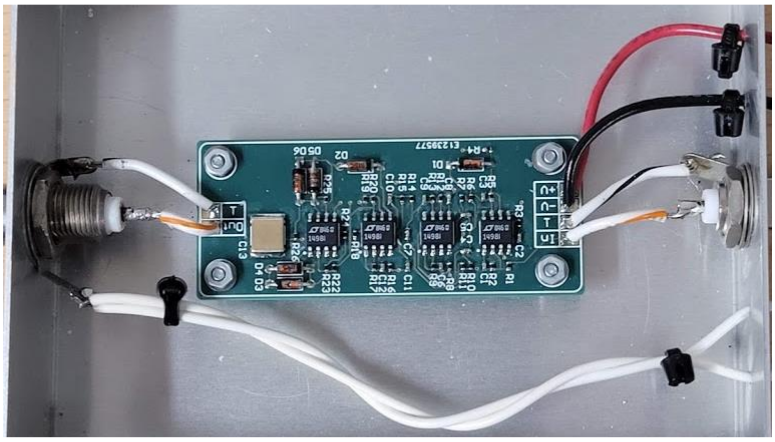
Figure 3 : instrument description

A pyroelectric crystal produces pulsed X-Ray. CSL (Centre Spatial de Liège) use a self-recalibration method during the measurement. This method is based on the monitoring of the fluorescence of a known small reference sample in the field of view of the instrument. This allows to monitor the flux of the source and hence renormalize continuously the spectrum during measurement to attenuate the source flux fluctuation's effects.

The detector is a Silicon Drift Detector (SDD) from Mirion as X-Ray detector for CSL device. The resolution given by the manufacturer is 127 eV (measured with manufacturer shaping amplifier).

The electronics were developed partly by CSL for the power supply and the analogue part, while the digital electronic was developed by KUL (University of Leuven). The analog part is a shaping amplifier to transform the signal coming from the detector-preamplifier module to Gaussian shape. It's a critical component and its capability is directly responsible of the spectral resolution of the whole instrument because the instrument resolution is the quadratic sum of the resolution of its detector and the resolution of the analog electronic readout.

The purpose of the digital electronic is to measure the amplitude of the Gaussian pulse at the output of the shaping amplifier. The amplitude of the Gaussian pulse is directly proportional to the energy of the detected X-Ray photon. The amplitude is obtained by a measurement of the pulse duration. This type of design is called Time to Domain MultiChannel Analyser (TDMCA).



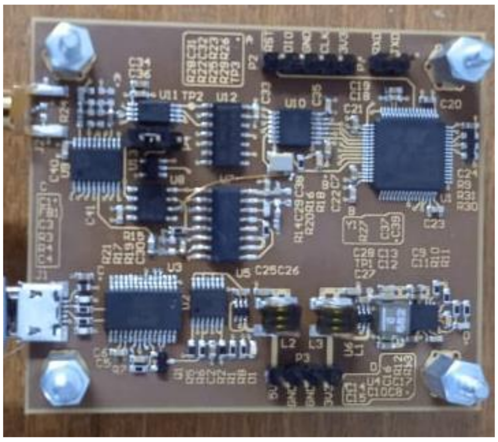


Figure 4 : Shaping amplifier and TDMCA realisation

3. Measurements

- Energy resolution

The critical parameter of an Energy Dispersive X-Ray Fluorescence (EDXRF) spectrometer is the energy resolution. It is measured by the width of the energy peaks. Better resolution is achieved with low value of Full Width Half Maximum (FWHM) for a given peak. Lower values allow a better S/N ratio in the energy spectrum and higher peak intensity in general.

Thanks to our analog electronic readout, we achieved a world record energy resolution of 121.17 eV as shown Figure 5**Error! Reference source not found.**. T he resolution of the latest Nasa instrument is 125 eV. Due to physics (quantum fluctuations in the charge generation process) the lower theoretical achievable resolution is 119 eV.

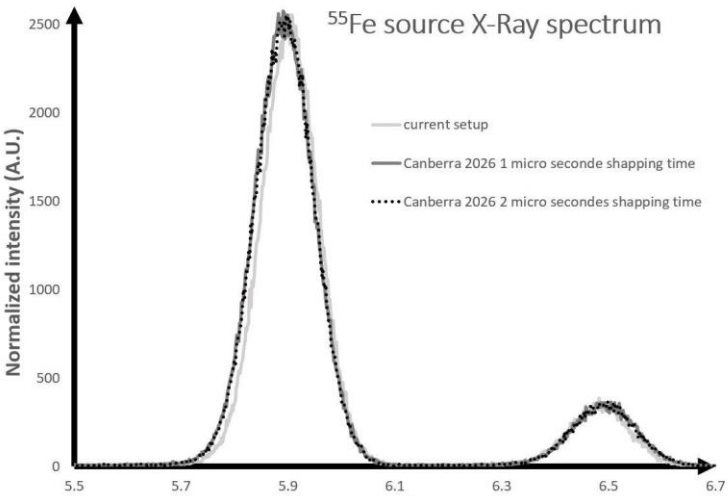


Figure 5 : World record energy resolution

- Spectrum renormalization

In portable XRF, as we are using pulsed PXG, variation within a pulse and inter-pulses variations are problematic.

Therefore, a process is introduced to monitor the total source emission during a measurement and normalize the measured spectrum for the total fluence of the source. The normalization factor is obtained by monitoring the fluorescence of a known reference sample in the field of view of the instrument. An Indium thin wire is used as reference to monitor the intensity of its L_α line at 3.28 keV. The relative standard deviation of the ten measurements, using the two PXG sources simultaneously (one with Cu target and one with Mo target), without renormalization was 25.2% and with normalization it was reduced to 3.71%.

- Peak selection algorithm

The Region Of Interest (ROI) width can be affected by the thermal environment and space radiation aging of electronic.

A peak search algorithm is used to define the best ROI for a given peak with a calibration. This could be implemented in flight with a filter wheel on board the instrument. The wheel would have several reference samples that would be used to re-run the algorithm to optimize ROI even with aging instrument in space ionizing radiation environment.

The wheel reference samples could be synthetic samples of anorthosite with increasing content of atoms of interest for lunar geology. The procedure is tested with samples of anorthosite with added amount of Mn (0.2%; 0,5%; 1%, 2% and 5%). The measured spectrum are given on the Figure 6. The algorithm is used with renormalized spectrum. The best ROI found with the algorithm has allowed to obtain a perfect linear calibration (r^2=1) as illustrated on the Figure 7. For higher concentration, a quadratic curve should be used to take into absorption or possible count reduction due to pileup.

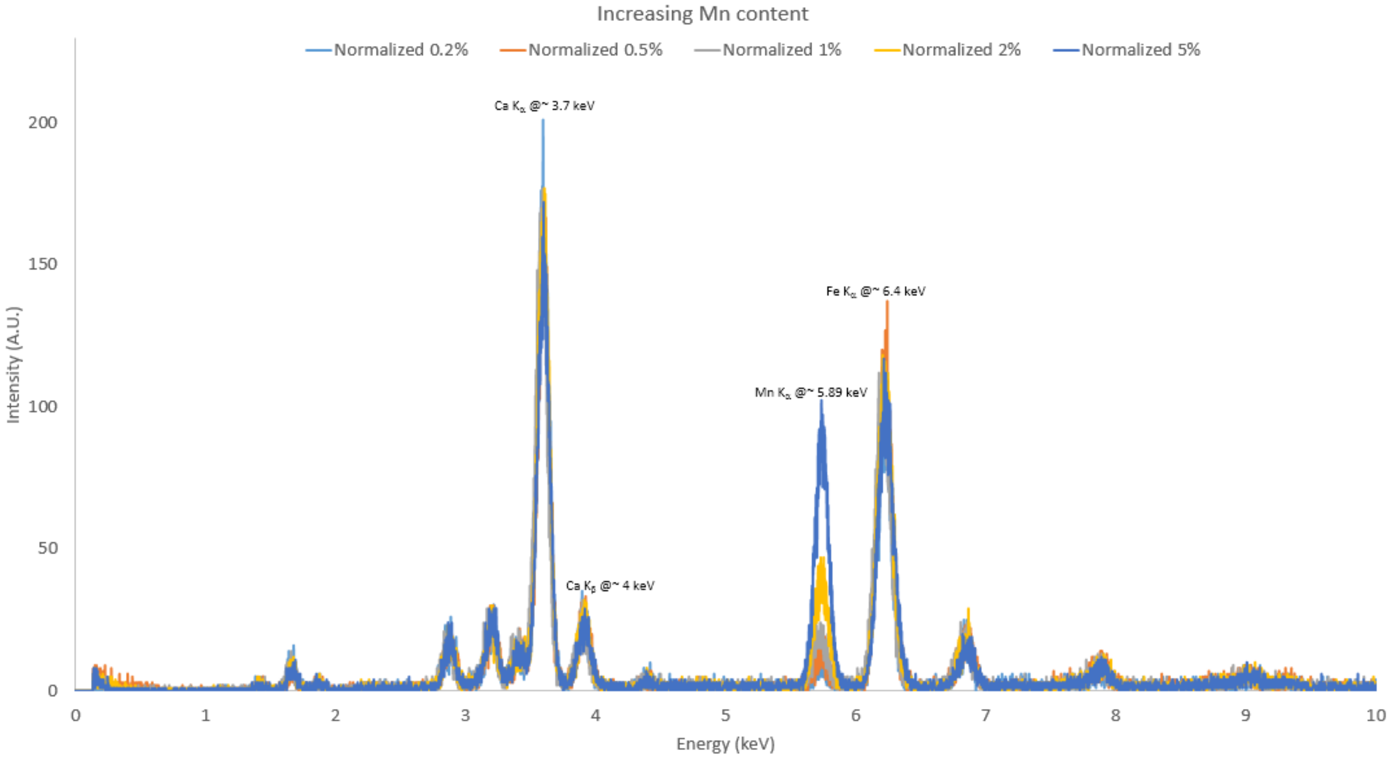


Figure 6 : Spectrum of anorthosite with increasing content of Mn

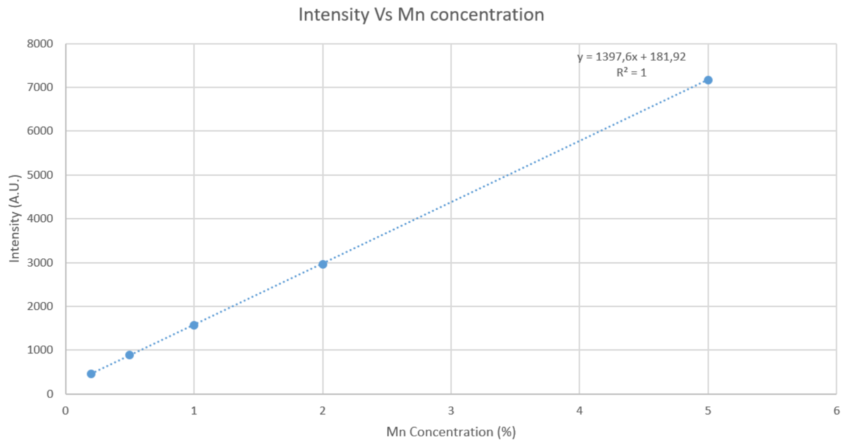


Figure 7 : XRF intensity versus concentration curve with peak selection algorithm

<u>- Measurement under vacuum</u>

To assess the instrument in a relevant environment, a vacuum test has been done.

The impacts of vacuum and low temperature on the measurement are:

- Low pressure increases the quality of the measurements, compared to measured made in air. First, the low energy X-Rays isn't absorbed by the air. Secondly, there is no fluorescence due to the argon present in the atmosphere.

- At low temperatures, it has been demonstrated that the resolution of SDD (silicon-drift detectors) is better. The X-Ray detector used in the XRF project is an SDD. The resolution of the measurement decreases (FWHM value, the lower is better) as the temperature decreases.

Anorthosite sample has been measured and the obtained spectrum is shown on Figure 8.

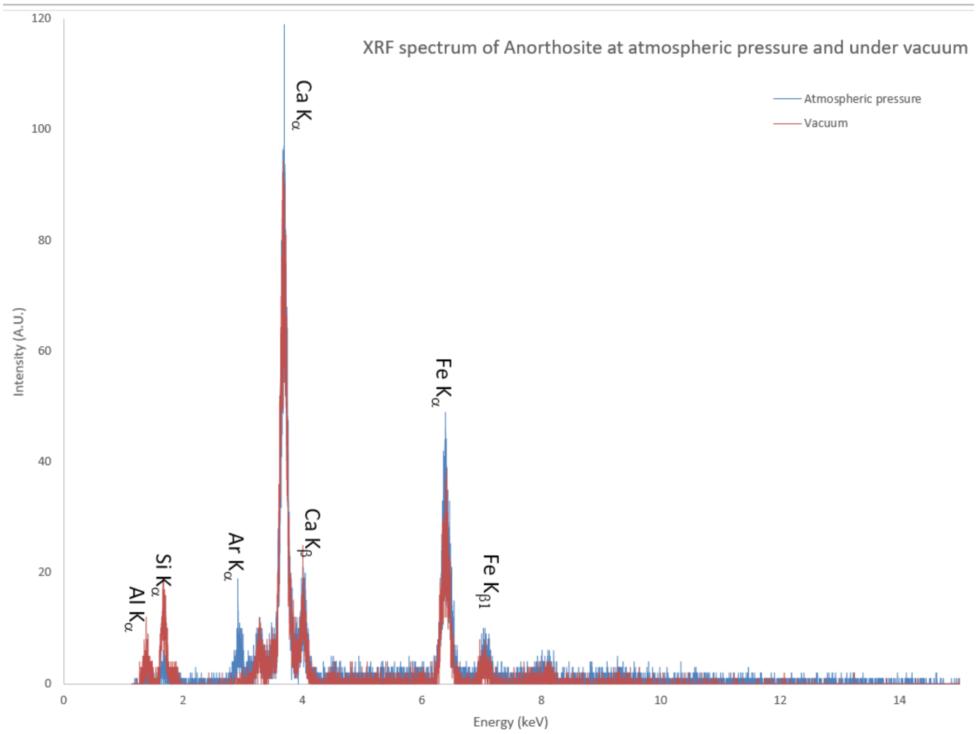


Figure 8 : Vacuum and atmospheric pressure comparison

The XRF instrument setup has been tested under vacuum in the Focal 1.5 vacuum chamber facility at CSL. Measurements were performed at different temperatures: 20°C, -20°C and 0°C.

The output signal from the shaping amplifier has shown degradation at ~ 5°C. The probable cause has been identified with theoretical simulation (LTSpice).

<u>- In-Situ Resources Utilization measurement</u>

Preliminary XRF measurements for monitoring In-Situ Resources Utilization (ISRU) process on the Moon for O_2 and metal production from regolith have been tested.

The spectrum is showed in the Figure 9 and for example, Figure 10 shows the XRF signal Vs TiO_2 content

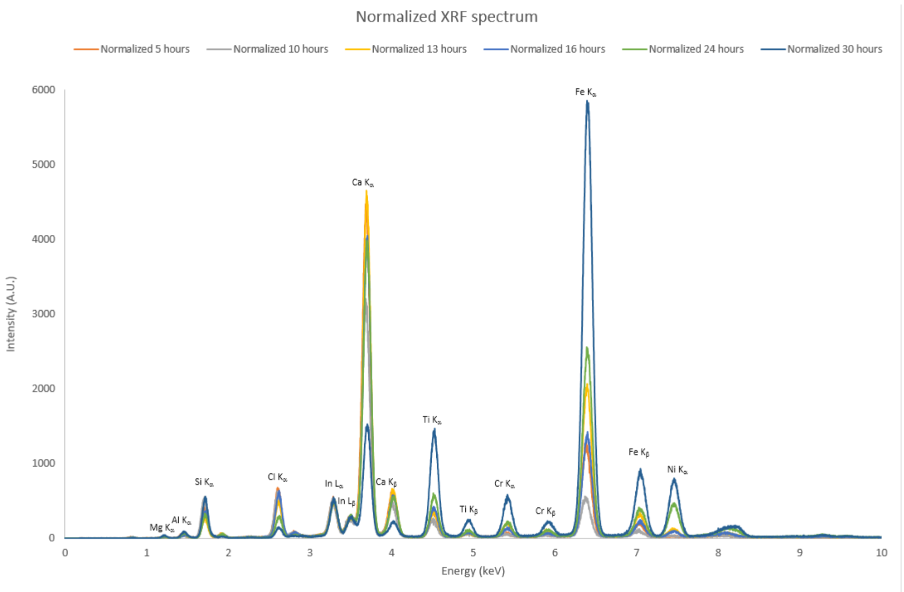


Figure 9 : XRF spectrum

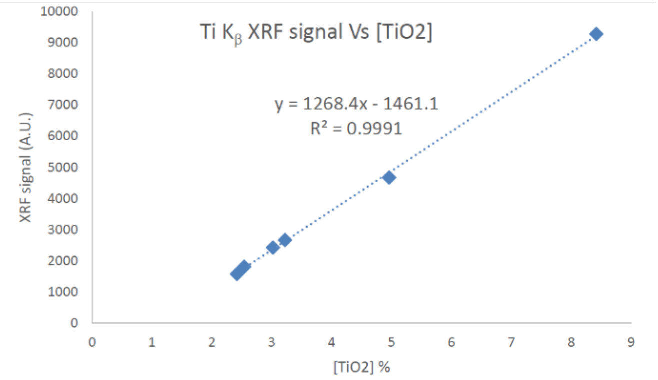


Figure 10 : XRF signal Vs Ti content

4. Conclusion

The XRF spectrometer developed in this project obtained an energy resolution of 121.17 eV, which is better than the NASA instrument which was limited to 125eV.

The sources used are no longer available but it is possible to replace them advantageously by Japanese sources.

The mode "free" space weather instrument" has been tested with our device when we turn the source OFF. If we want to implement this mode, additional calibrations are needed at several energies and with several type of particle/radiation (γ rays, protons, electrons …)

Successful preliminary ISRU monitoring measurements have been done.

We have therefore developed an instrument that can be used for different types of measurement in both space and ground applications.

This project was made possible thanks to the contribution of KUL, MAGICS, MIRION, BIRA/IASB, ULiège.