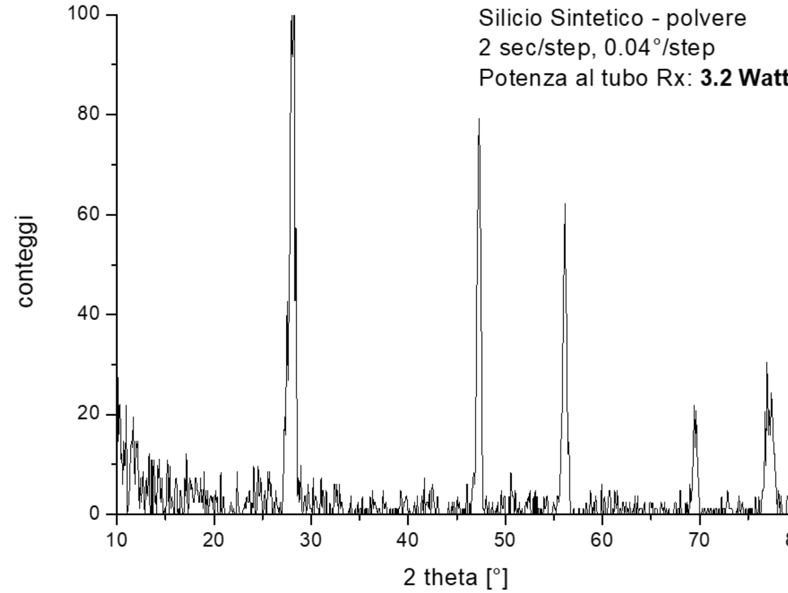
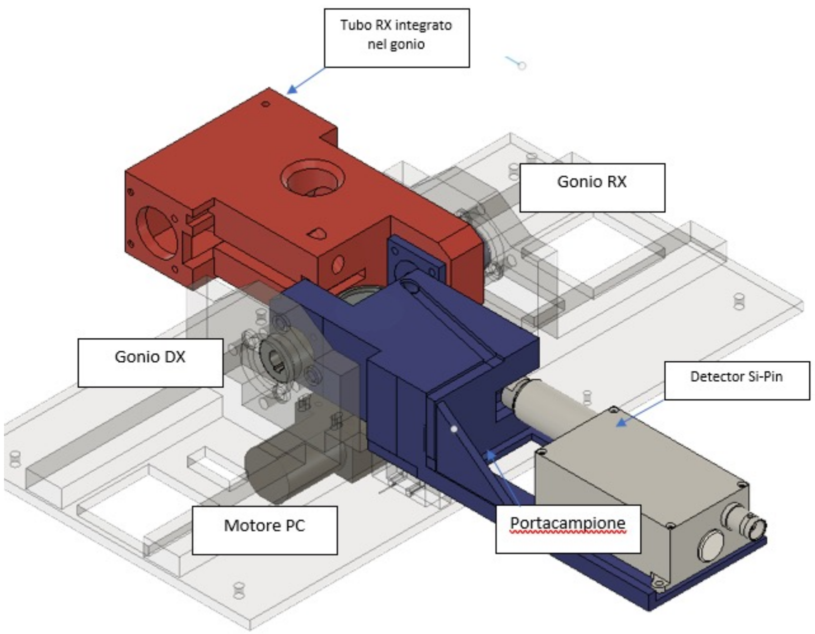
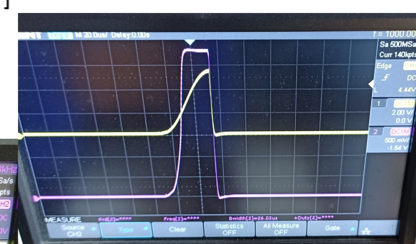
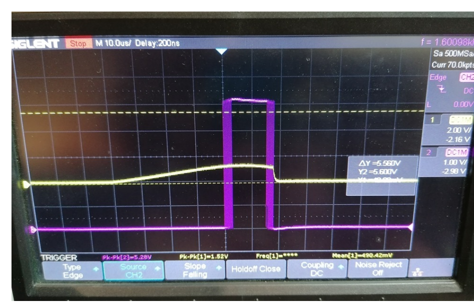
A new way to develop a LOW COST instrumentation: the **PICO RX diffractometer/XRF spectrometer, enhanced with polycapillary lenses**

Sultan Dabagov[1], Dariush Hampai[1], Paolo Plescia[2], Manuel Volakakis[3]









1: iNFN XlabF
2: CNR Ist. Geologia Ambientale e Geoingegneria
3: NC Technologies Spa

“A new way to develop a **LOW COST" instrumentation: the PICO RX diffractometer/XRF spectrometer** enhanced with polycapillary lenses

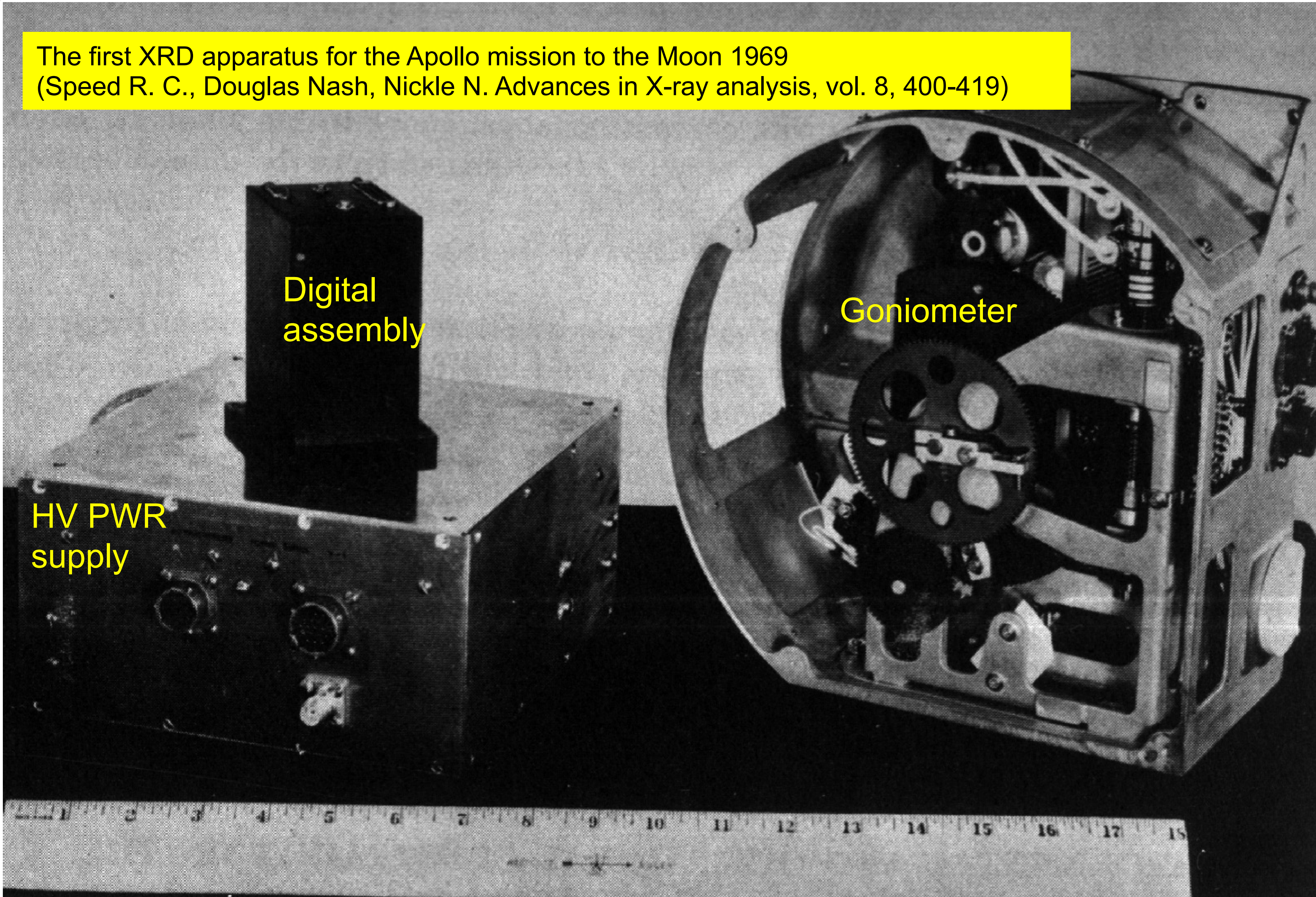
A short introduction

The work presented here is the sign of the beginning of a broad public-private collaboration in the X-ray sector, in particular in the field of the application of polycapillary RX optics, of which INFN is leader. These polycapillary optics made at the INFN XlabF in Frascati will be used to make instruments belonging to two specific categories:

- the **PICO series**, **low-cost instruments**, easily transportable and cheap to maintain, but equipped with RX polycapillary lenses
- the **RXR series**, **high-end instruments**, equipped with the best X-ray collimation, detection, imaging and data collection technologies

The brief introduction that will follow is the description of the main steps taken to create the instruments of the PICO series, retracing the main stages in the history of portable instruments in Italy, starting from the instruments of the early 2000s onwards.

The first XRD apparatus for the Apollo mission to the Moon 1969
(Speed R. C., Douglas Nash, Nickle N. Advances in X-ray analysis, vol. 8, 400-419)



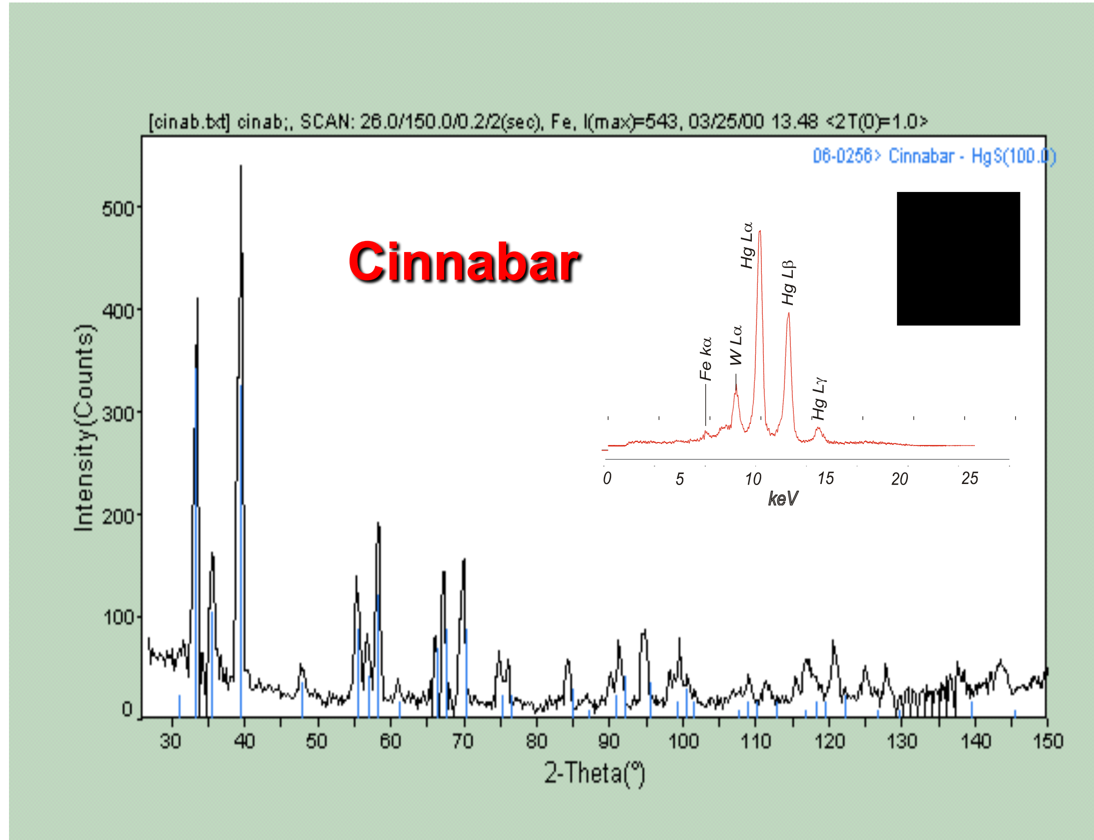
these pioneering instruments

Portable instrumentation: an Italian Hystory

X-ray Portable instrumentation: an Italian Hystory

The first combined XRF/XRD portable X-ray instrument for applications in the world of cultural heritage was born in 1996: it was called ASX (or "sexi") and used detector part of the Mars ROVER technology (Amptek XR100CR).
it was produced by ASSING SpA of Monterotondo with which the CNR had a collaboration agreement and of which I was the scientific leader

X-ray Portable instrumentation: an Italian Hystory



1998: DUST - The first commercial portable X-ray diffraction and X-ray fluorescence instrument. It was equipped with a Bragg-Brentano geometry goniometer, step motors, Si Pin detector and slit collimation system and a laser rangefinder for the right sample focus

X-ray Portable instrumentation: an Italian Hystory

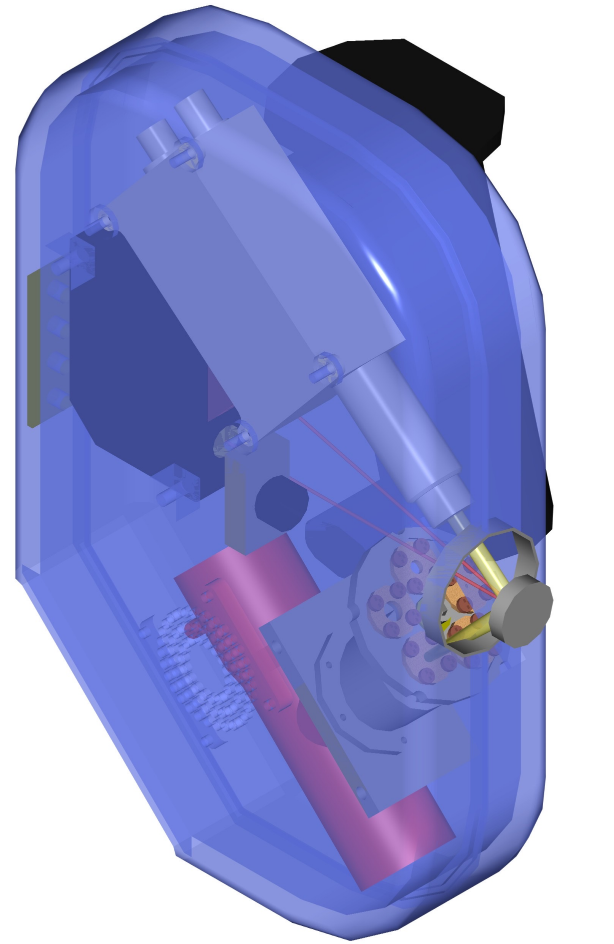
The XRF spectrometer LITHOS, the first «italian», commercial, XRF portable spectrometer appear in **2003**

EDS resolution of 160 eV, Peltier cooling

Laser interpherometer (± 5 mm)

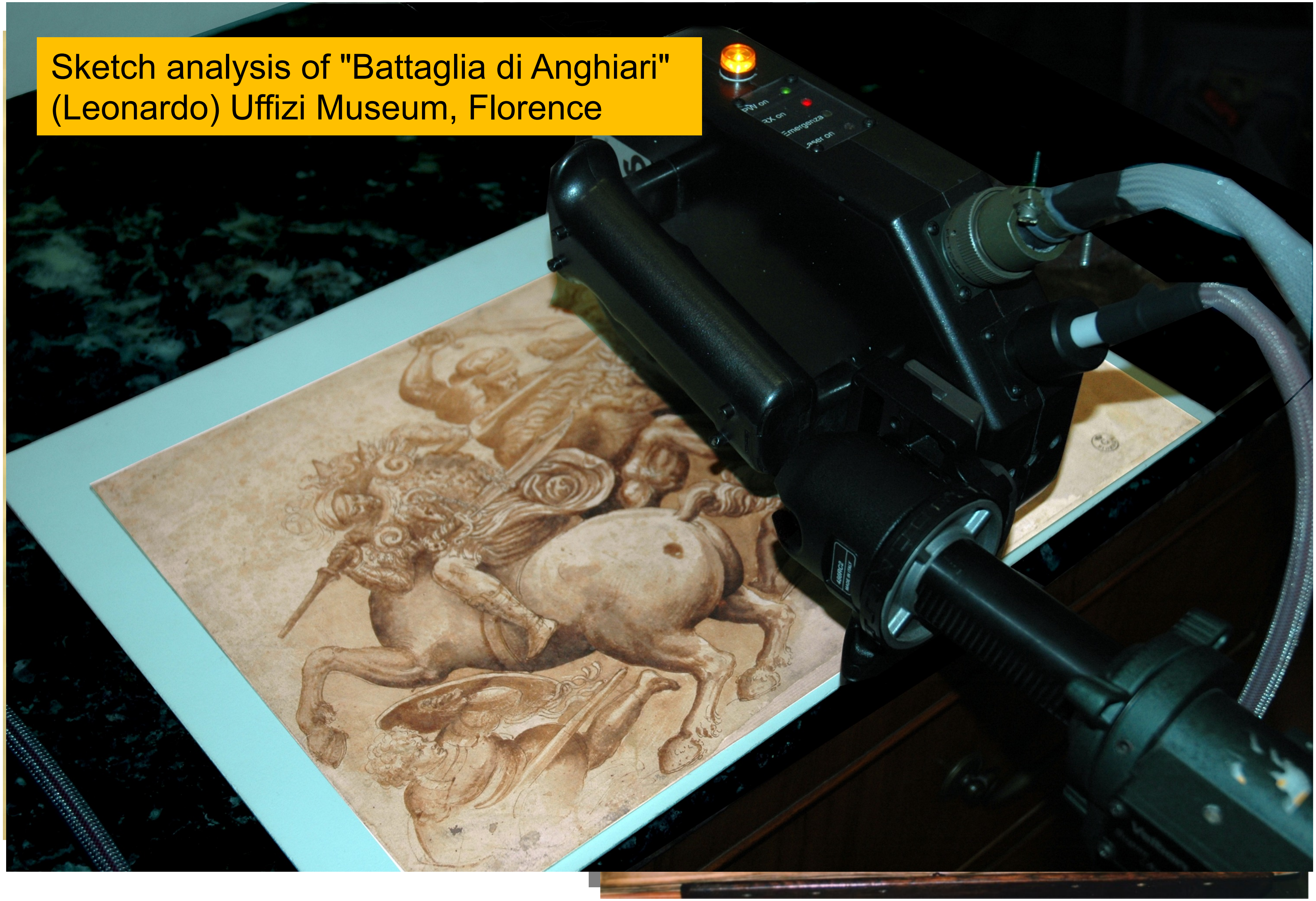
X-ray optics composed by 5 collimators, 1 monochromator and five filters

An X ray tube with a Mo anode, 30 kV- 0.5 mA

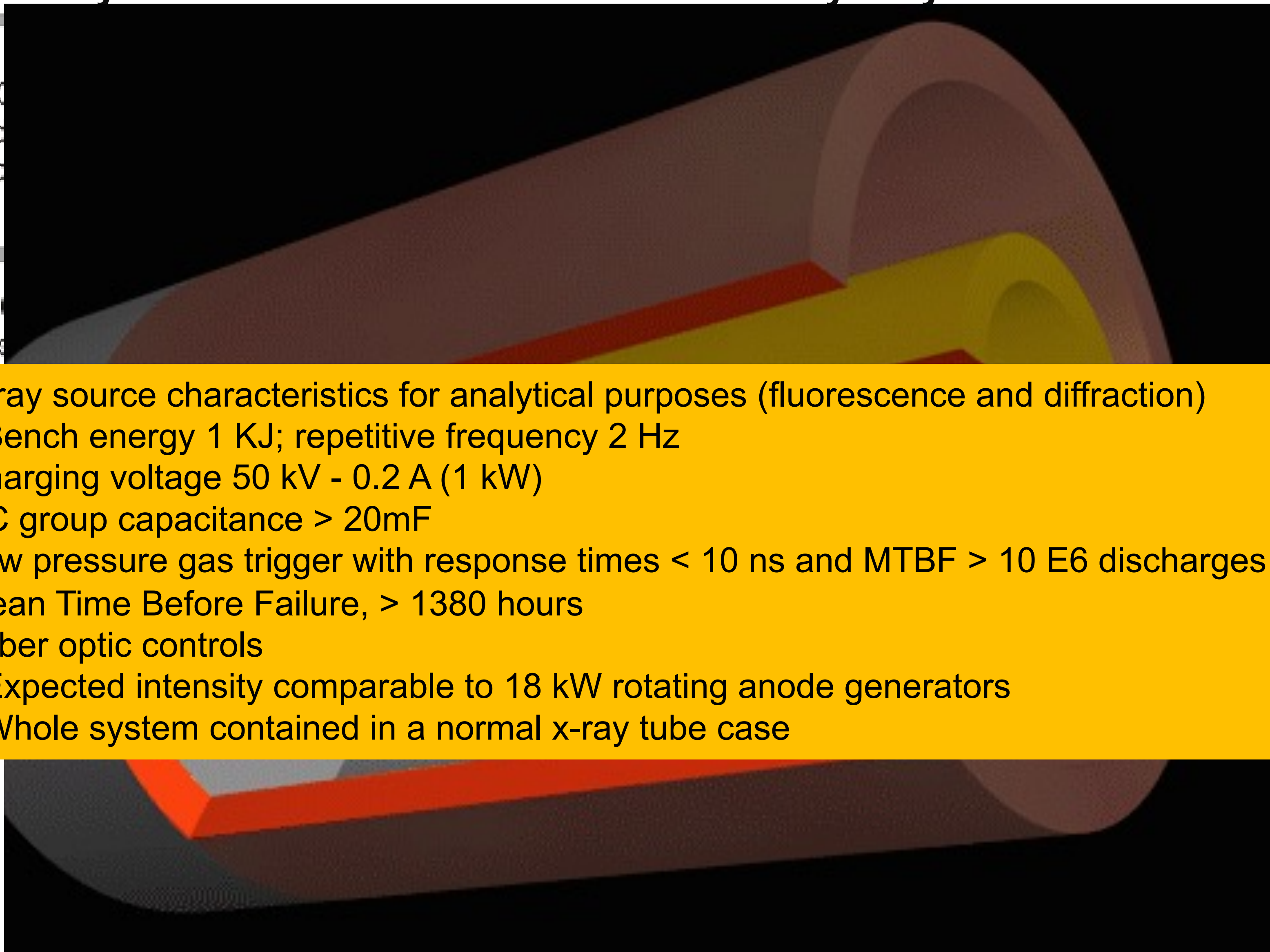


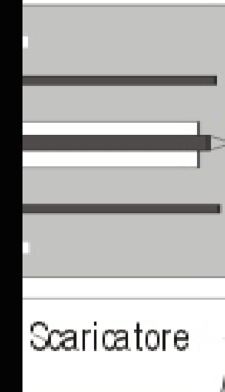
X-ray Portable instrumentation: an Italian Hystory

Sketch analysis of "Battaglia di Anghiari"
(Leonardo) Uffizi Museum, Florence



X-ray Portable instrumentation: an Italian Hystory





X-ray source characteristics for analytical purposes (fluorescence and diffraction)

· Bench energy 1 KJ; repetitive frequency 2 Hz

Charging voltage 50 kV - 0.2 A (1 kW)

RC group capacitance > 20mF

Low pressure gas trigger with response times < 10 ns and MTBF > 10 E6 discharges

Mean Time Before Failure, > 1380 hours

· fiber optic controls

· Expected intensity comparable to 18 kW rotating anode generators

· Whole system contained in a normal x-ray tube case

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e source

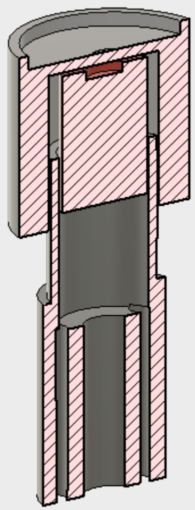
X-ray Portable instrumentation: a NEW Italian Hystory

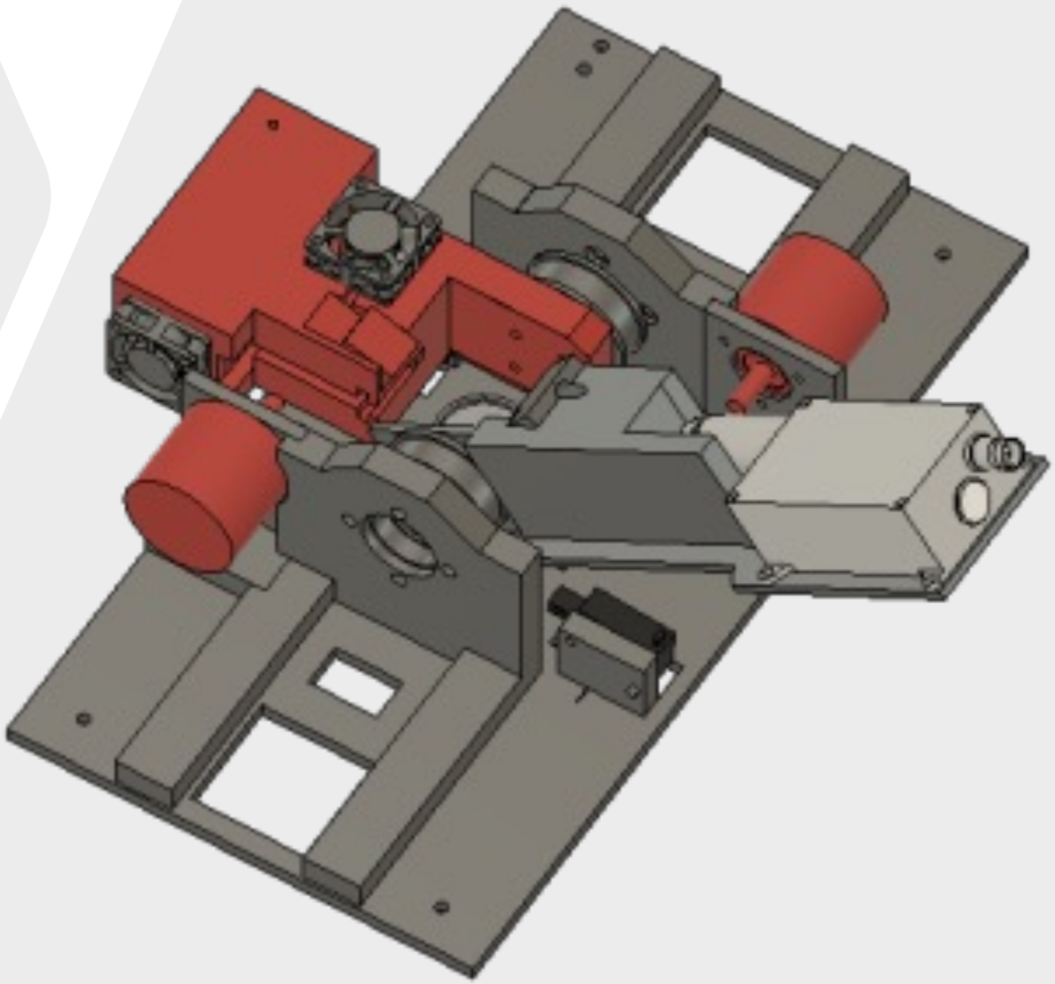
In 2022 a new phase begins in the development of RX instruments suitable for a wider and more varied public, by overcoming the problem of costs and poor analytical sensitivity. The idea of the authors of this work is to create tools that cover the market segment that has so far not had access to X-ray tools, such as restoration laboratories, medium-sized museums, private laboratories dealing with environmental hygiene and of work, professionals in mining research, mineralogy and gemology, professional studies and many others.
The idea is to bring X-ray diffraction to become the main "speciation" technique in all these areas.
The new project, conducted between the INFN polycapillary laboratory, NC Technologies and proven technical consultants, aims to overcome the main problems encountered in the previous activities and in particular:

- ***The brightness of the X-ray sources*** →solution: polycapillary lenses

- ***Instruments and spare parts costs*** → solutions: reduced costs by "open source" software, 3D printed hardware in carbon fiber and PLA, latest generation electronics, based on ARDUINO type MCU and programmable analog/digital PSOC platforms. 88% of the materials of our instruments is completely recyclable

Inside PICO





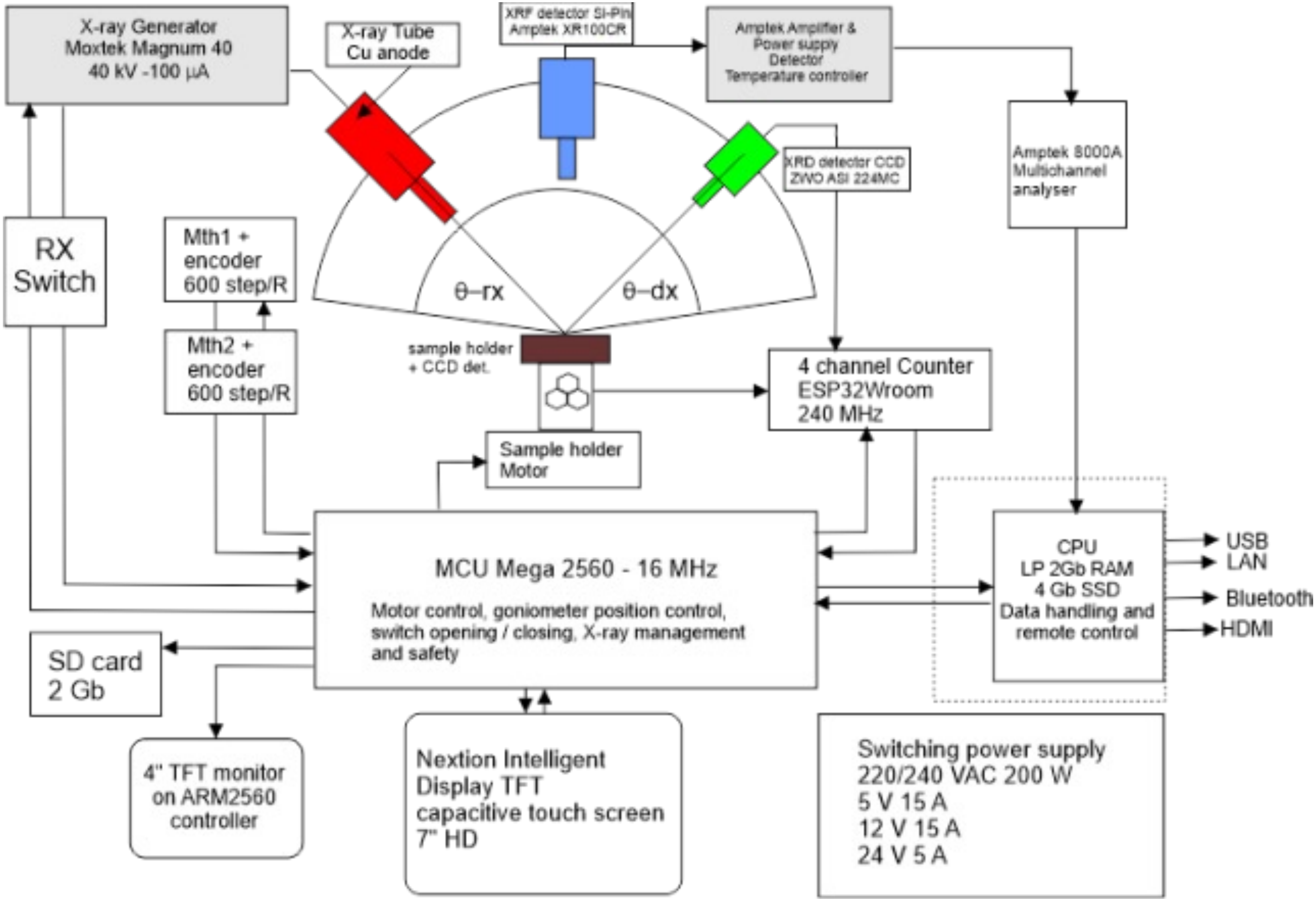
The PICO XRD X-ray diffraction analyzer

The low cost of the tool should not make one think of a lower quality: the instrument is built using network resources and minimizing construction costs by means of 3D printing.

The electronics and the software have been created using OPEN SOURCE platforms, such as in particular: MCU of the ARDUINO type and software based on Python and Processing, whose code is easily transformable and upgradeable by the User.

An innovative aspect of the instrument is the use of FDM 3D printing for the creation of many parts of the system, such as the fixed parts of the goniometers, the arms and almost 80 % of the instruments structure.

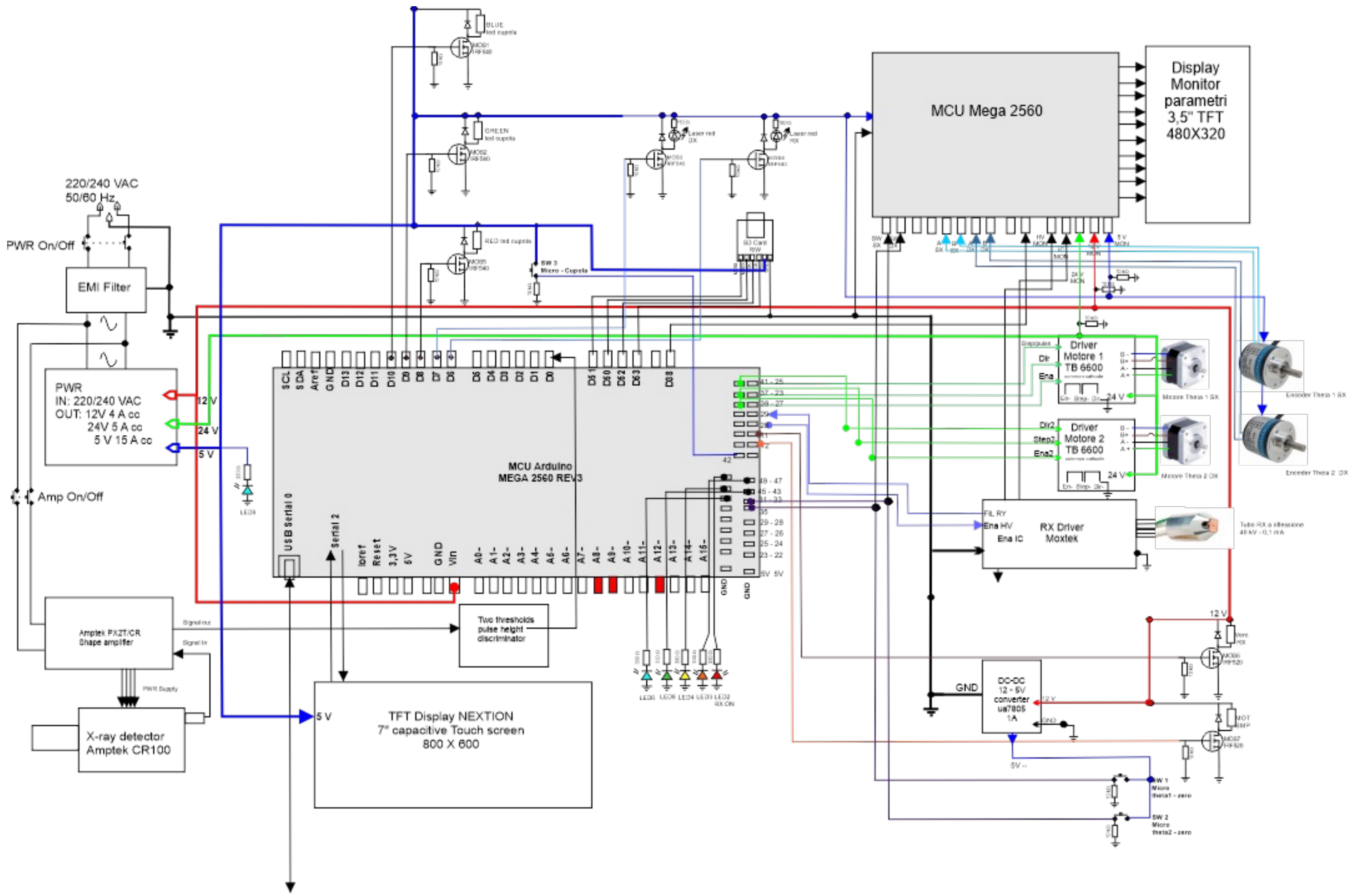
The low power guarantees low emissions: in normal working conditions (1.0 h of acquisition at maximum power, with the lid down) it emits, at a distance of 10 cm from the chassis, an average of 0.2 mSv/h; considering the legal limit for exposure of the skin of professionally exposed people, equal to 500 mSv/year, the instrument emits 0.35% of the permitted dose.



PICO: HOW IT’S DONE

PICO: HOW IT’S DONE

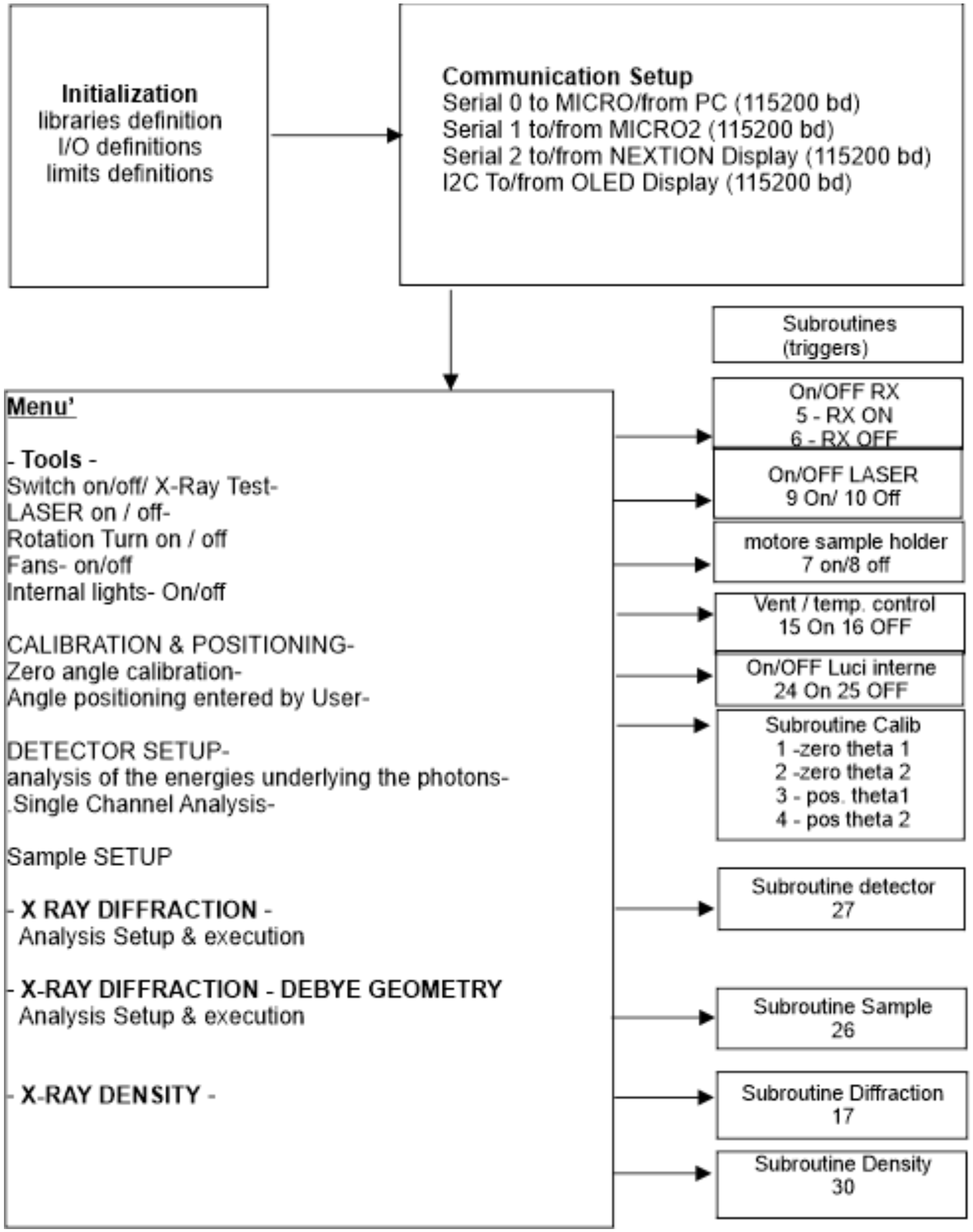
PICO XRD: ELECTRIC DIAGRAM

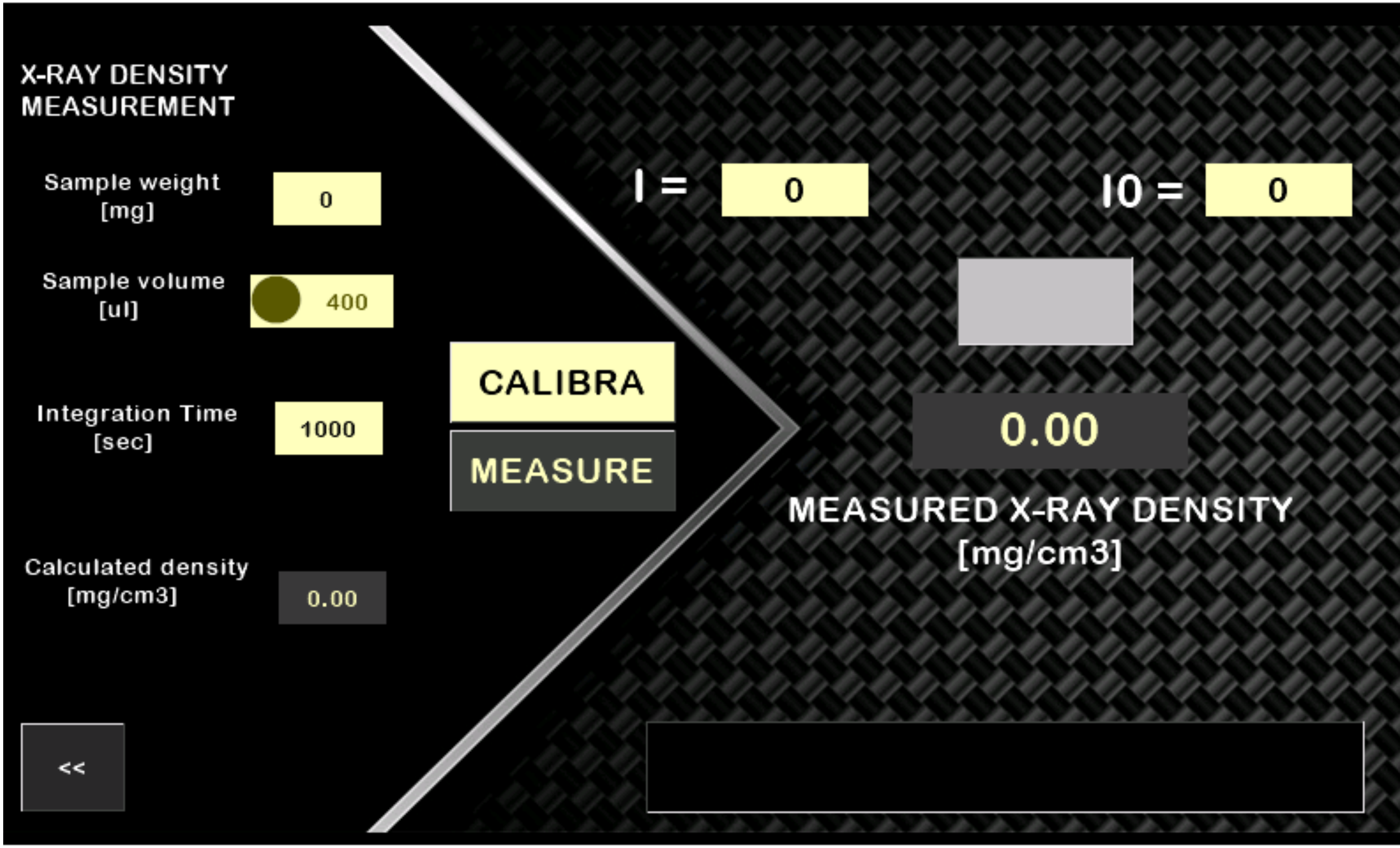


PICO is built on a Theta-Theta Bragg-Brentano geometry, with independent axes; tube and detector are mounted on goniometric arms which have an action range between 0 and 65° of theta angle, i.e. between 0 and 130° 2-theta

PICO: HOW IT’S DONE

Software of PICO series
The PICO series software is entirely made in Python to be compatible with ARDUINO Microcontrollers. The programming of the various modules was also carried out thanks to the help of the AI (Chat GPT Plus), in particular in the definition of the best algorithms for analyzing the signal coming from the detector and discriminating only the component coming from the diffraction of the k alpha anodic line. Thanks to this approach, the amount of lines of code required has been reduced by more than a third.



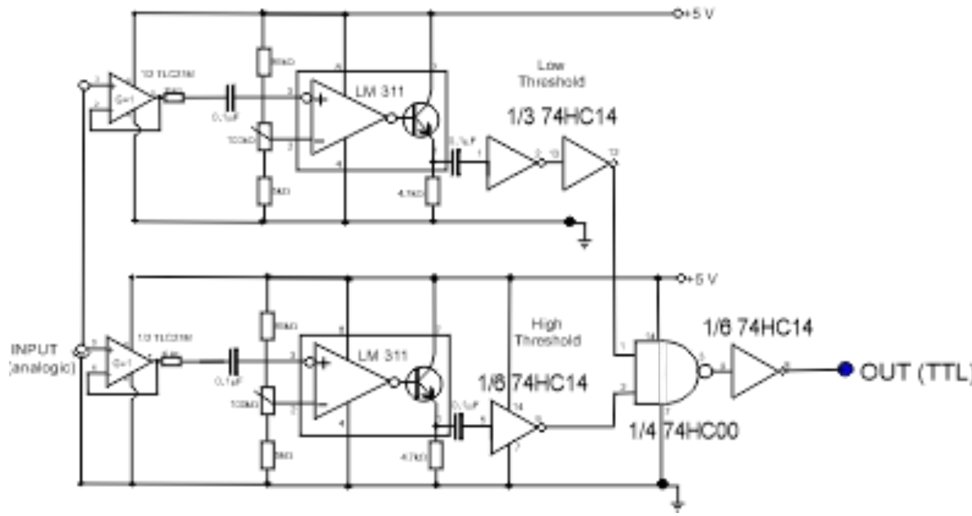

X-RAY DENSITY MEASUREMENT
Sample weight [mg]
0
Sample volume [ul]
400
Integration Time [sec]
1000
Calculated density [mg/cm3]
0.00
CALIBRA
MEASURE
I =
0
I0 =
0
0.00
MEASURED X-RAY DENSITY [mg/cm3]
<<

PICO: HOW IT'S DONE

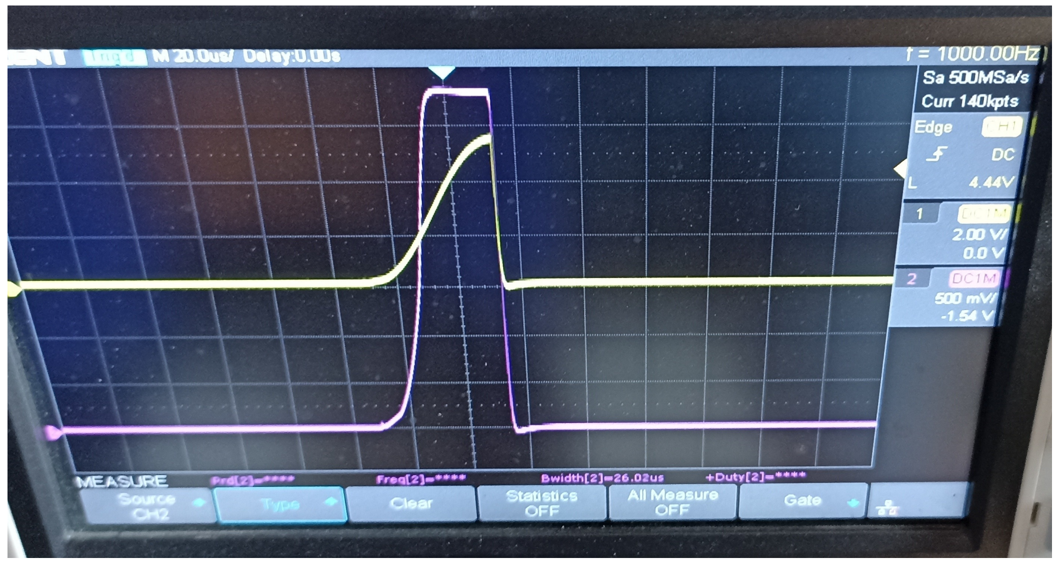
Development of electronics suitable for the management of Cu-ka radiation from Si-Pin detectors

detector → Shaping Amp → Window comparator + anticoincidence → MCU

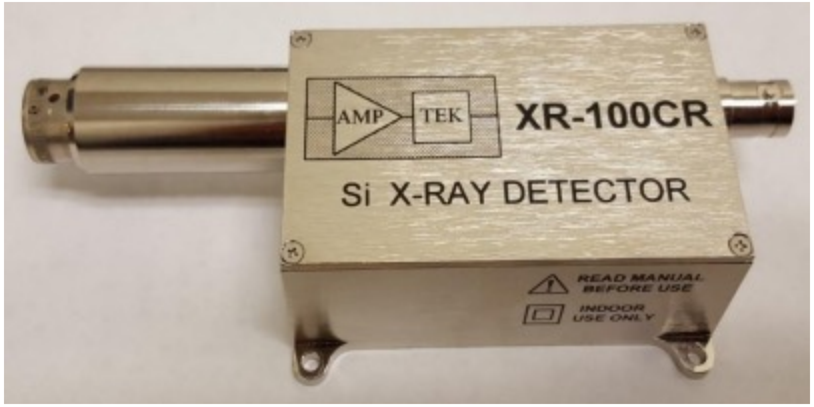
Two Threshold pulse height discriminator



The signal coming from the shape amplifier (px2cr by Amptek) with a duration of about 20 us, is squared by an anti-coincidence circuit and subsequently fed to an ARDUINO DUE MCU, equipped with the Atmel SAM3X8E ARM Cortex-M3- 32 Bit processor which it allows to perform a high resolution AD conversion and therefore to discriminate the various energy amplitudes of each pulse train.



PICO: HOW IT'S DONE



X-RAY SOURCES

The X-ray generation system is built by the American company Moxtek and consists of a Side-end tube and a microgenerator. The 4 W power is generated using a voltage of 40 kV (maximum) and a maximum current of 100 uA. The tube is directly inserted into the goniometer arm (first theta axis), equipped with two cooling fans to ensure a sufficiently low surface temperature. The arm carrying the X-ray tube is equipped with a parallelizing optics, with two slits with divergence of 1°. The same optics will be replaced by a polycapillary semi-lens to obtain a perfectly parallel beam on the sample

X-RAY DETECTOR

The detector is a Si-Pin diode from the American Amptek, the Si-Pin XR 100 CR detector with a 20 micron beryllium window and Peltier thermoelectric pump cooling. Obviously it is also an energy dispersive spectrometer (EDS), with a resolution on the ka line of Mn of 160 eV. The detector is powered by its Amptek PX2CR power supply amplifier.
The detector arm is equipped with optics similar to that of the RX arm-tube, with the possibility of mounting two slits from 0.3 to 1° to focus the beam from the sample. There are no plans to adopt polycapillary optics on this segment of the goniometer

PICO: HOW IT'S DONE

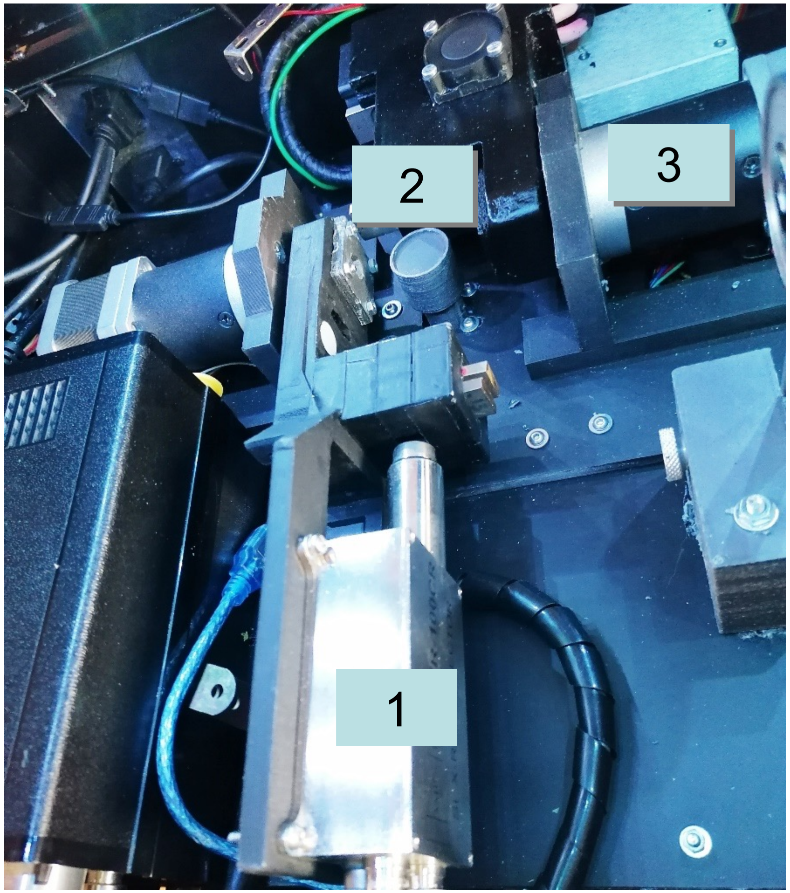
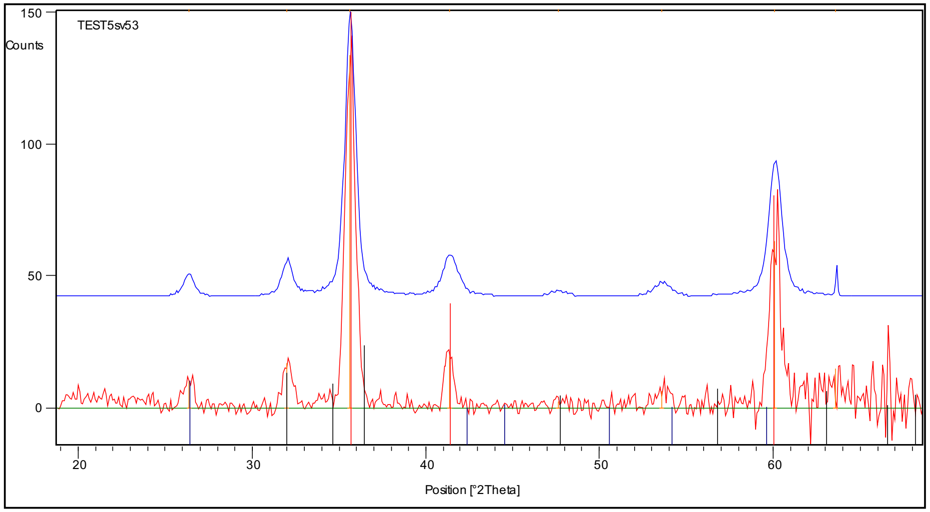


Image of the sample camera of PICO: detector (1), sample holder (2), stepper motors (3))

First prototype of PICO in 2022

XRD Tests on materials

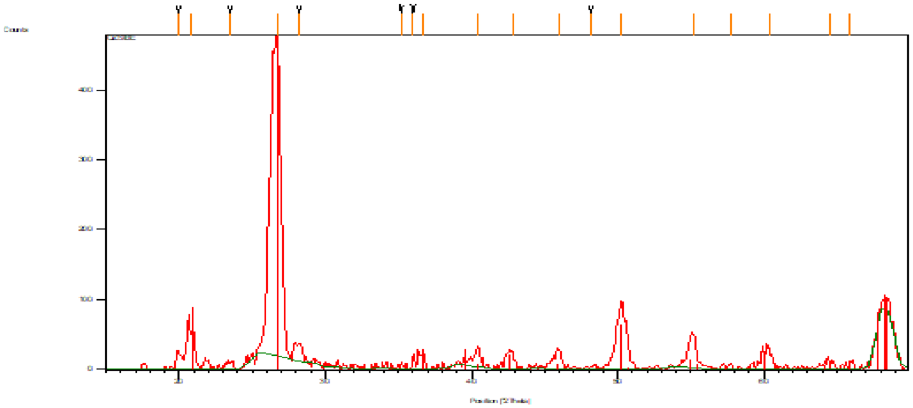
Silicon carbide (synthetic): qualitative analysis



Pos. [-2Th.]	Height [cts]	FWHM [-2Th.]	d-spacing [Å]	Rel. Int. [%]
26.3548	10.24	0.7085	3.37900	7.66
32.0006	16.86	0.7085	2.79456	12.60
35.6149	133.75	0.7085	2.51881	100.00
41.3971	19.41	1.0627	2.17936	14.51
47.6294	2.36	1.0627	1.90772	1.77
53.5856	6.43	1.0627	1.70887	4.81
60.0637	63.29	0.8856	1.53912	47.32
63.5712	14.95	0.1123	1.46238	11.18

Ref. Code	Score	Compound Name	Displacement [-2Th.]	Scale Factor	Chemical Formula
01-073-1708	54	Moissanite 3\ITC\RG, syn	0.200	1.074	Si C
00-029-1131	35	Moissanite 6\ITH\RG, syn	0.200	0.947	Si C
01-075-1621	7	Graphite 2\ITH\RG	0.200	0.017	C

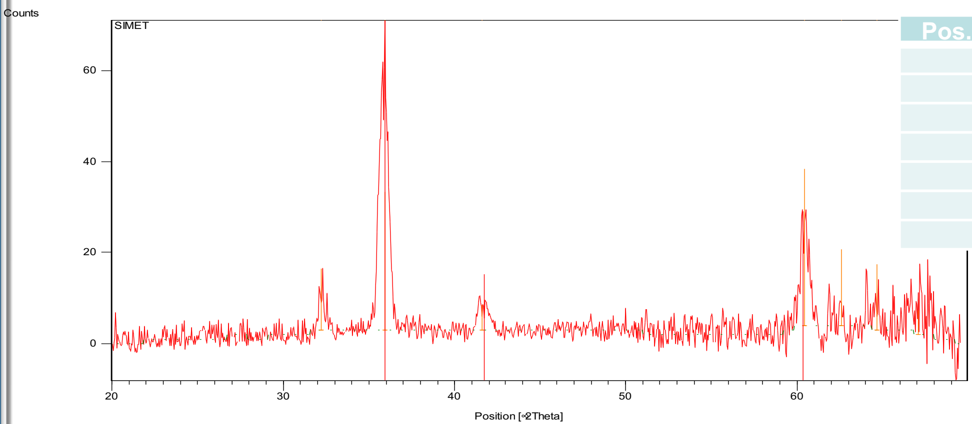
Quartz sand: qualitative analysis - sample not grounded



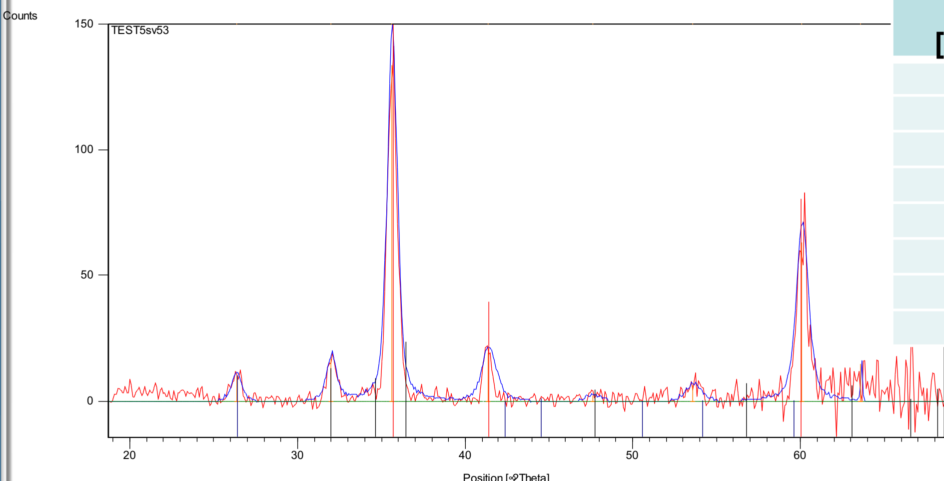
Peak List

Pos.[°2Th.]	Height[cts]	FWHM[°2Th.]	d-spacing[Å]	Rel.Int.[%]
19,9278	29,67	0,2657	4,45557	6,45
20,8060	60,84	0,3542	4,26945	13,23
23,4913	7,42	0,5314	3,78714	1,61
26,7325	459,93	0,8856	3,33487	100,00
28,1922	23,70	0,3542	3,16543	5,15
35,1647	4,65	0,3542	2,55213	1,01
35,9891	19,51	0,2657	2,49554	4,24
36,7180	22,25	0,3542	2,44765	4,84
40,3890	29,19	0,3542	2,23325	6,35
42,7942	27,05	0,3542	2,11314	5,88
45,9462	27,84	0,4428	1,97525	6,05
48,1667	1,43	0,5314	1,88925	0,31
50,1677	77,89	0,4428	1,81849	16,93
55,1800	42,89	0,4428	1,66458	9,32
57,7093	5,70	0,2657	1,59750	1,24
60,3496	31,29	0,2657	1,53378	6,80
64,5272	8,21	0,8856	1,44420	1,79
65,8214	3,54	0,6480	1,41773	0,77

PICO: Diffraction test (without poly Lens)



Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]
32.2273	13.53	0.2492	2.77772	39.22
35.9170	30.51	1.4170	2.50038	88.45
41.5731	5.65	0.8856	2.17234	16.39
60.4158	34.49	0.4396	1.53226	100.00
62.5937	16.76	0.1569	1.48408	48.60
64.6406	14.40	0.1555	1.44194	41.76
67.0602	5.30	1.2960	1.39452	15.35



Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]
26.3548	10.24	0.7085	3.37900	7.66
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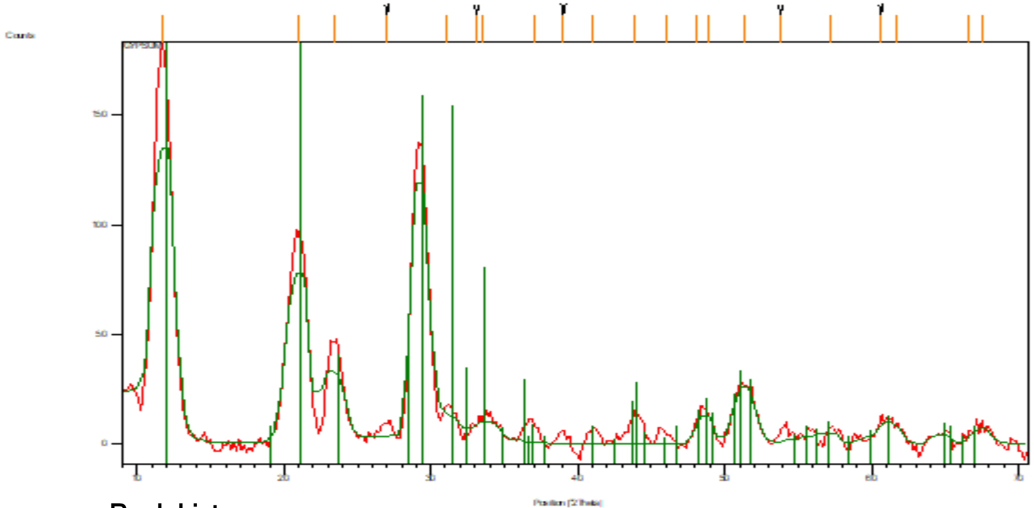
60.0637	63.29	0.8856	1.53912	47.32
63.5712	14.95	0.1123	1.46238	11.18

XRD of synthetic Silicon Carbides from a ceramic production line

Ref. Code	Score	Compound Name	Displacement [°2Th.]	Scale Factor	Chemical Formula
01-073-1708	54	Moissanite 3\ITC\RG, syn	0.200	1.074	Si C
00-029-1131	35	Moissanite-6\ITH\RG, syn	0.200	0.947	Si C
01-075-1621	7	Graphite 2\ITH\RG	0.200	0.017	C



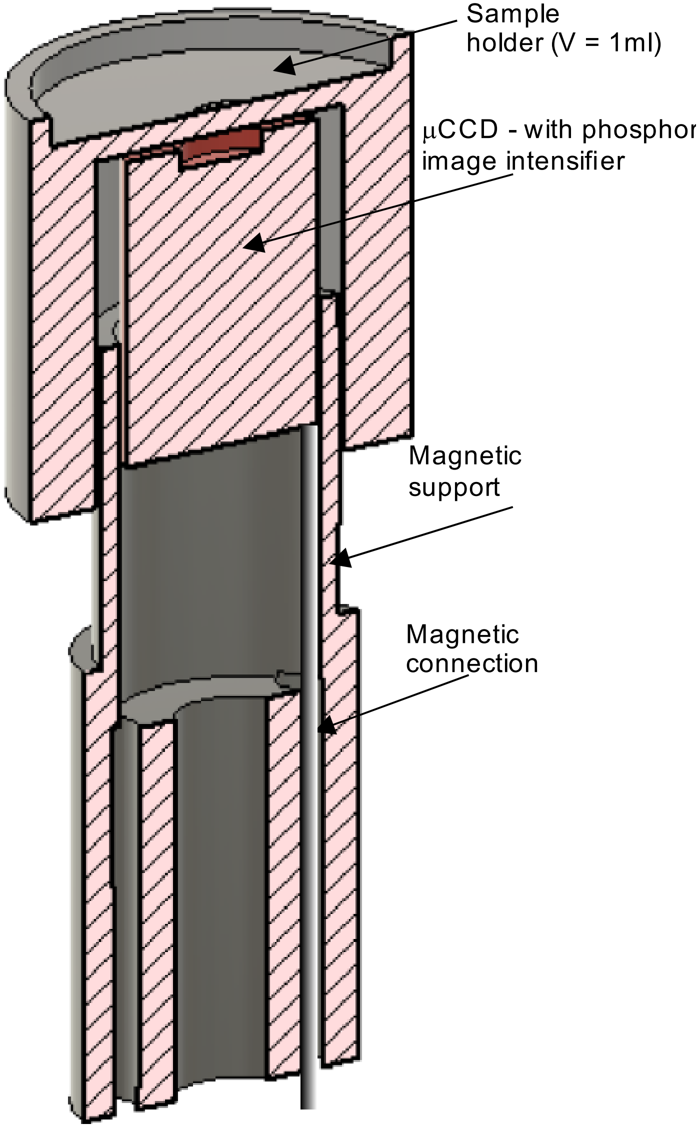
PICO: Diffraction test (without poly lens)

XRD analysis of gypsum powder and indicization of peaks

Peak List

Pos.[°2Th.]	Height[cts]	FWHM[°2Th.]	d-spacing[Å]	Rel.Int.[%]	Tipwidth[°2Th.]	Matched by
11,7431	108,98	1,3565	7,53614	100,00	1,6278	00-021-0816
21,0099	50,89	1,4399	4,22848	46,70	1,7278	00-021-0816
23,4726	50,43	0,5260	3,79011	46,28	0,6312	00-021-0816
27,0052	5,08	0,7085	3,30180	4,66	0,8502	
31,0753	9,30	0,6654	2,87801	8,53	0,7985	00-021-0816
33,1057	1,00	0,0900	2,70599	0,92	0,1080	
33,5394	12,10	0,5335	2,67199	11,10	0,6402	00-021-0816
37,0160	6,23	0,8856	2,42863	5,72	1,0627	00-021-0816
39,0246	7,71	0,7085	2,30813	7,08	0,8502	
40,9927	7,93	0,7085	2,20175	7,27	0,8502	00-021-0816
43,9057	13,80	0,7085	2,06219	12,67	0,8502	00-021-0816
46,0124	4,51	0,7085	1,97256	4,14	0,8502	00-021-0816
48,0479	3,65	0,0900	1,89364	3,35	0,1080	00-021-0816
48,9480	1,00	0,0900	1,86091	0,92	0,1080	00-021-0816
51,3531	12,28	0,7265	1,77926	11,26	0,8718	00-021-0816
53,8130	6,26	0,8640	1,70218	5,75	1,0368	
57,2296	3,00	0,0900	1,60975	2,75	0,1080	00-021-0816
60,6503	6,65	0,0900	1,52689	6,10	0,1080	
61,7305	3,17	0,0900	1,50274	2,91	0,1080	00-021-0816
66,5916	2,83	0,0900	1,40436	2,59	0,1080	00-021-0816
67,4918	6,00	0,0900	1,38780	5,51	0,1080	00-021-0816

PICO Accessories



- **Active sample holder** with a CMOS detector, to measure thikness and density of sample

- **Debye sample holder:** sample holder with shape suitable for the Debye diffraction geometry.
In this sample holder, the sample is placed in a glass capillary with a diameter of 0.1 to 1 mm, which is "wet" by the X-ray beam collimated on a spot of 100 microns in diameter. the sample is rotated at the standard speed of 30 RPM.

PICO: Comparisons with industrially produced instruments of the same class (Competitors)

The only competitor currently on the market is the Olympus diffraction system which is present with two systems, BTX III and Terra II, they are diffractometric analyzers suitable for the rapid determination of crystalline phases and their quantification. The instruments we are talking about have relatively high costs (between 65 and 75 k€) and a low resolution, due to the lack of an angular scan. They use a Debye geometry with direct detection of diffraction patterns on a CCD. On the other hand they have an high reading speed.

	TERRA	PICO
Risoluzione spettrale 2 θ	0,25°	**0,01°**
Range analitico 2 θ	5 – 55 °	**0 – 90°**
Tipo detector	Peltier cooled CCD	Peltier Cooled Si-pin
Risoluzione XRF	200 eV @ 5.9 KeV	**160 eV** @ 5.9 KeV
Range energetico XRF	3 – 25 KeV	**3 – 35 KeV**
Xray tube voltage max	30 kV	40 kV
X – ray power max	10 W	4 W
Data storage	40 Gb	4 – 12 Gb
Connettività	Wireless	USB, Wireless (opt)
Peso	14,5 kg	18 kg

Thank you for your attention ..