ZnSe crystals for 0vDBD experiments: production and quality assurance issues

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LUCIFER

made in the frame of LUCIFER experiment
FP7/2007-2013
ERC grant agreement n. 247115.

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outline

- introduction

  (dedicated materials are needed for the next generation of 0vDBD experiments)
- crystals for 0vDBD experiments
  - large scale production issues
  - materials for scintillating bolometers
- crystals for LUCIFER experiment

  (production and certification)
- concluding remarks

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introduction

- extremely low interaction with the matter
- experimental constraints:
    - very low background
    - extremely high sensitivity

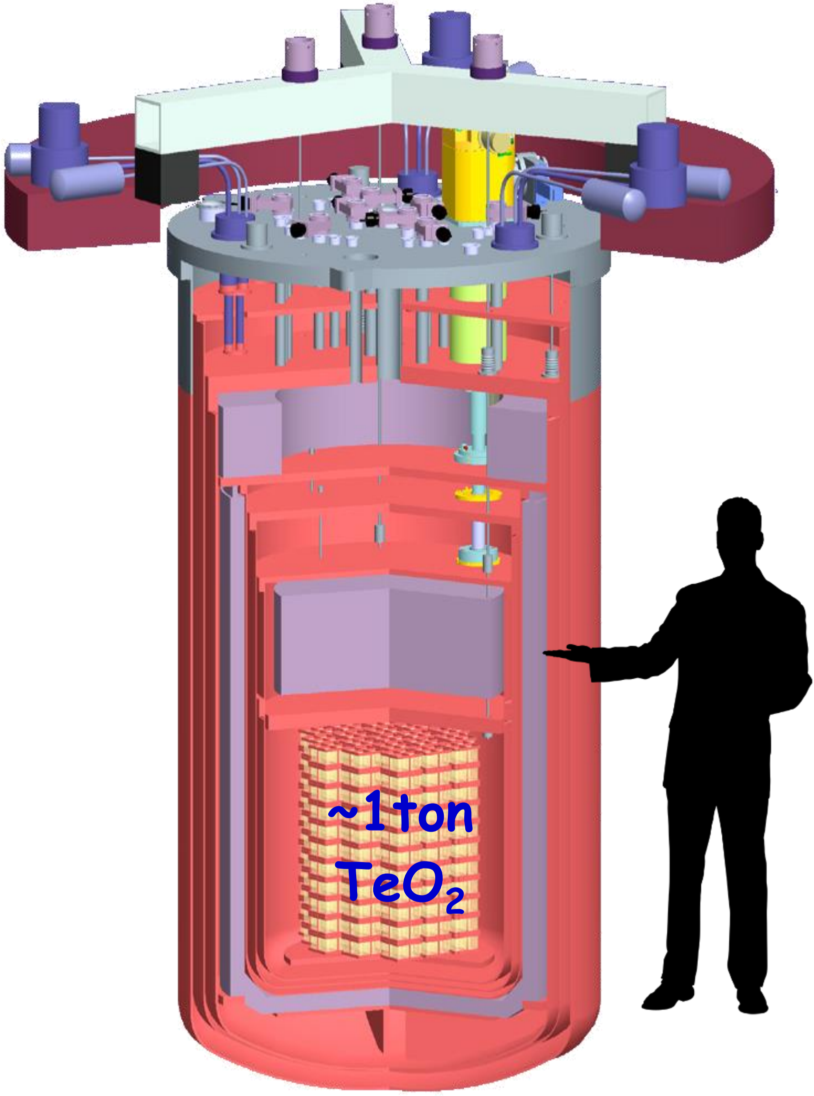
satisfying these two conditions is not enough

- new detectors are needed
- main (technological) constraint:
    - radio-purity of materials used

dedicated materials are needed to built increasingly sophisticated detectors for the next generation of 0vDBD experiments

large scale production issue

semantics "large scale"



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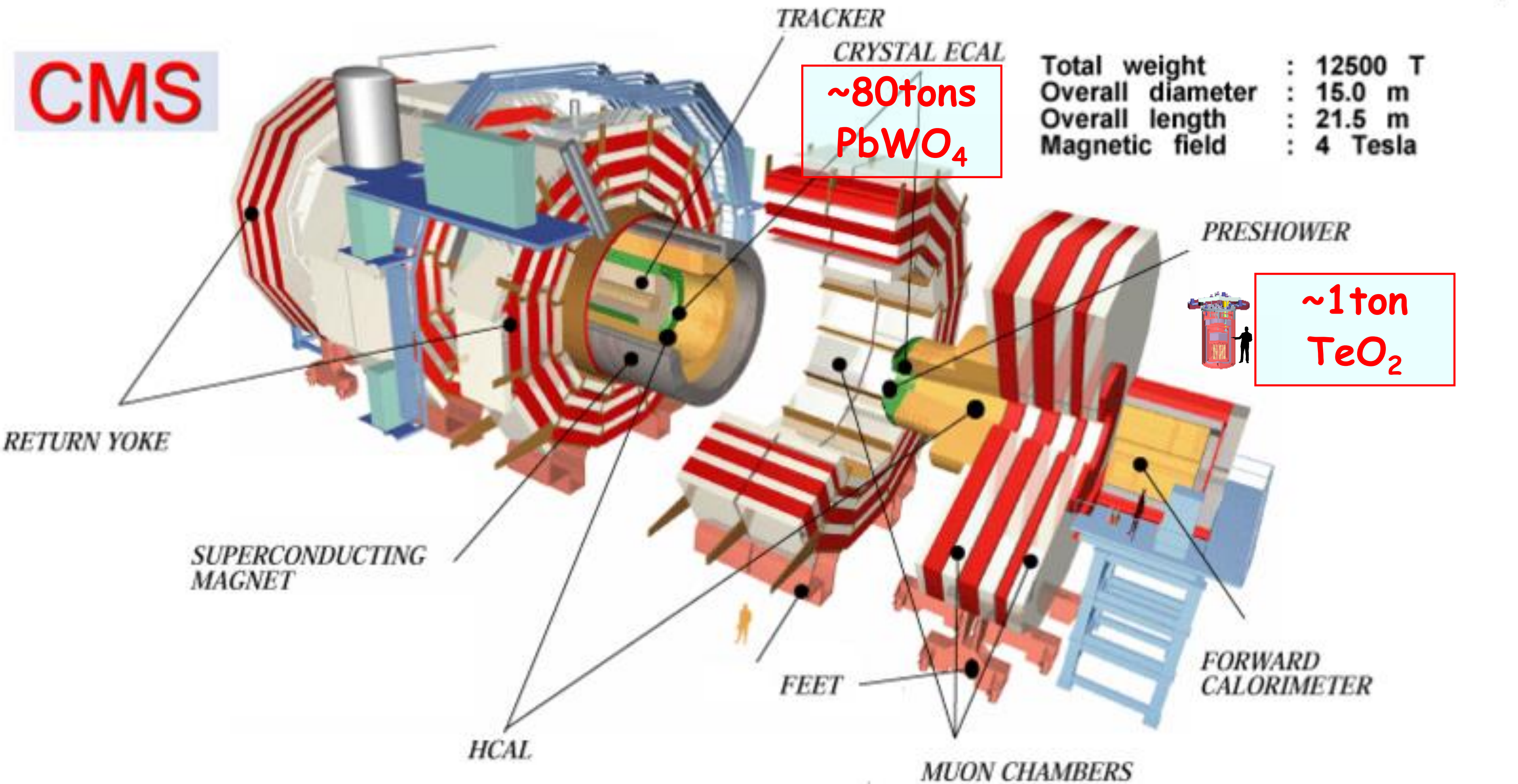

Compact Muon Solenoid

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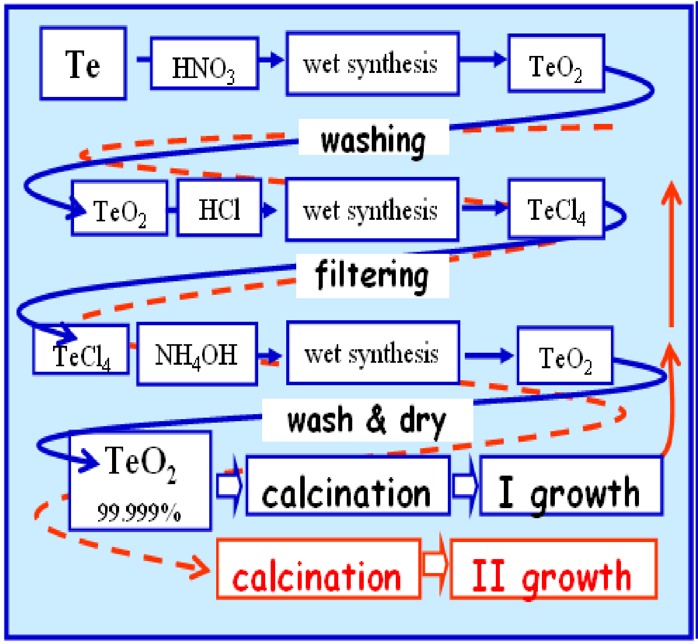
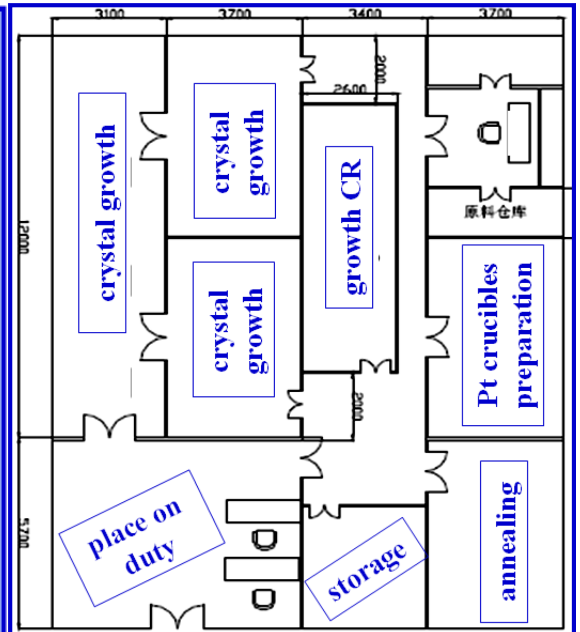
CUORE, first (successful) case

only 2-3% crystals can be measured

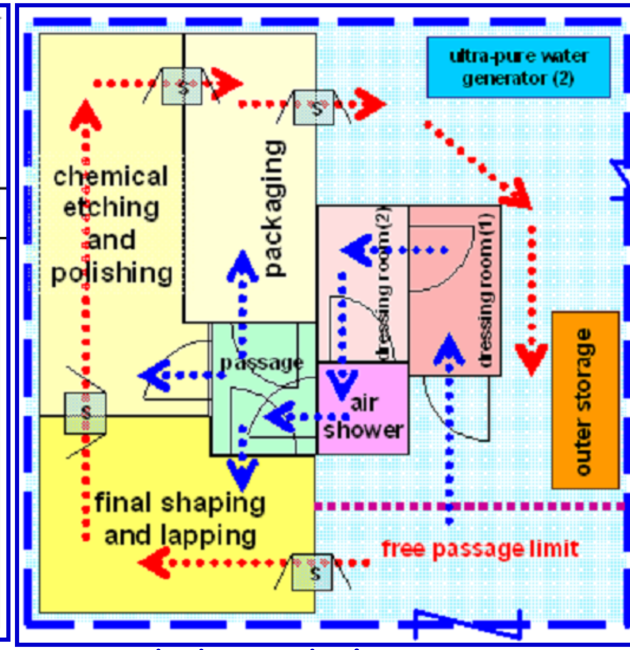
precautionary principle: raw materials, reagents, intermediary products, processes and procedures



dedicated TeO2 synthesis facility @ Kunshan Chemical Plant



dedicated crystal growth facility @ SICCAS Jiading



dedicated clean room SICCAS/INFN @ Jiading

dedicated production protocols
- environment conditions
- equipment and infrastructures
- materials handling

dedicated measurement protocols
- dimensions
- crystal perfection
- radio-purity

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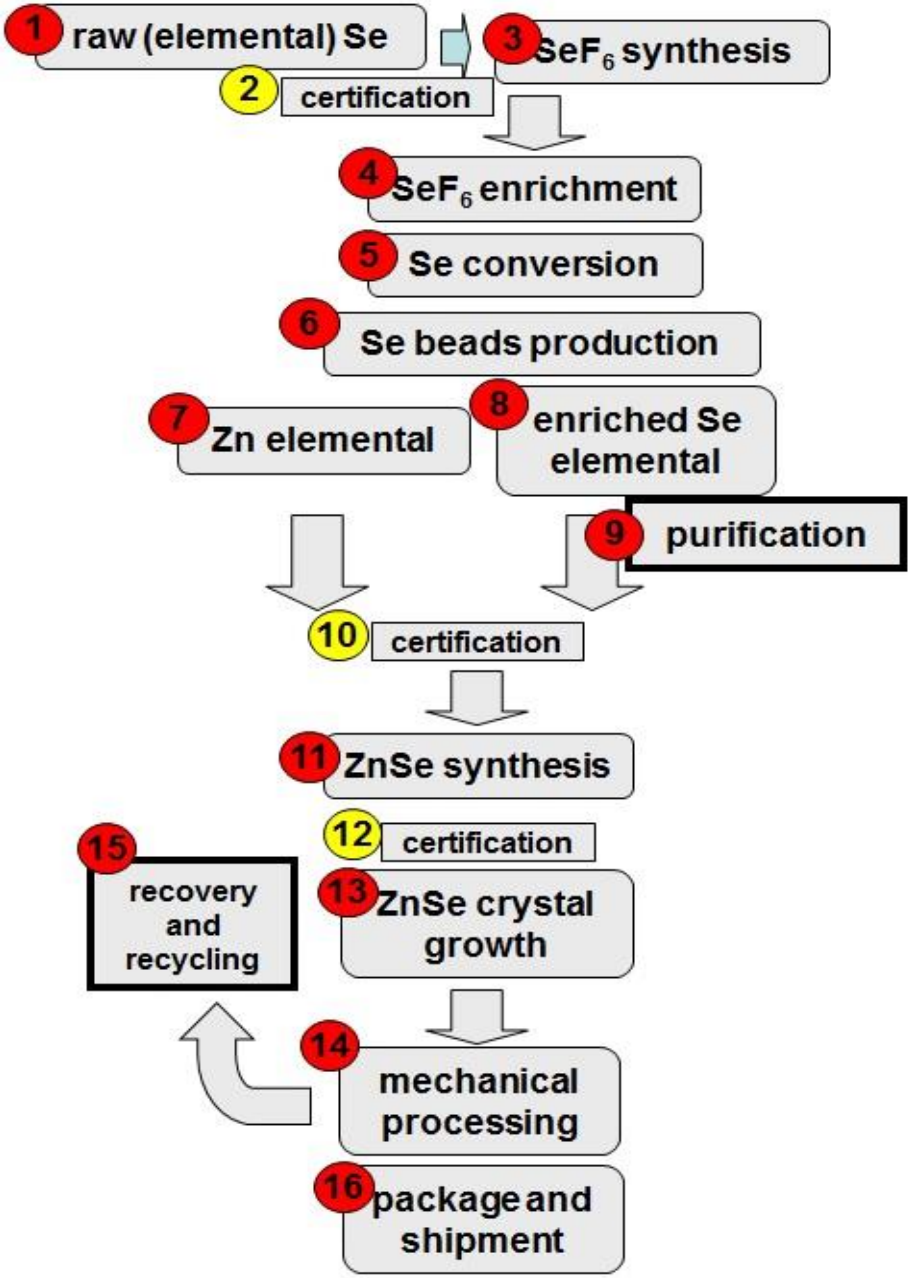
- **question:**
  - is it feasible a similar enterprise for 0vDBD experiments of next generation?
- **answer:**
  - TeO2 crystal was a lucky case:
    - industrial scale production already existed
    - easy implementation of improvements imposed by DBD application
    - long term existing market for high quality crystals
    - certification was (practically) needed (only) to guarantee the radio-purity
  - enriched/scintillating crystals is tricky case:
    - no scintillating material for DBD use was produced at large scale till now
    - some of the problems to be addressed were solved in the case of TeO_2 crystal production
    - the elevated price of the raw material makes very complicate each production step
    - need to start from elemental raw materials
    - certification needed not only for radio-purity

ZnSe crystal production



2014, November 14

- **4** completed
- **5** completed
- **8** in schedule — non distilled beads are distilled now
- **9** solved problem — purification through sublimation on the way, 4kg were sent back for distillation
- **11** solved problem — setup commissioned and method consolidated at ISMA
- **13** important progress — ISMA or ISC : production place still to be decided

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ZnSe crystal production timeline*

	$Zn^{82}Se$ crystals production plan (september 2014)								
y/w	month	days	ready to be delivered or processed	^{82}Se (purified) [kg]	^{82}Se (purified) TOTAL [kg]	^{82}Se synthesized to ZnSe [kg]	^{82}Se synthesized TOTAL [kg]	$Zn^{82}Se$ crystals [pcs]	$Zn^{82}Se$ crystals TOTAL [pcs]
2014/19	May	5-9	9May14						
2014/23	June	2-6	6Jun14	1.000	1.000				
2014/27	July	30-4	4Jul14						
2014/31	August	28-1	1Aug14						
2014/38	September	15-19	19Sep14	1.500	2.500	HPGe test of purified ^{82}Se			
2014/44	October	27-31	31Oct14	2.000	4.500				
2014/48	November	24-29	29Oct14	2.000	6.500				
2014/51	December	15-19	19Dec14	2.500	9.000	3	3		
2015/5	January	26-30	30Jan15	3.000	12.000	3	6	start crystal production	
2015/9	February	23-27	27Feb15	3.000	15.000	3	9		
2015/13	March	23-27	27Mar15	end ^{82}Se production		3	12	4	4
2015/17	April	20-24	24Apr15			3	15	4	8
2015/21	May	18-22	22May15			end ZnSe synthesis		4	12
2015/25	June	15-19	19Jun15					6	18
2015/29	July	13-17	17Jul15					6	24
2015/32	August							6	30
2015/40	September							6	36
2015/44	October					end crystal production			
2015/49	November								
2015/53	December								
2016/	January								
2016	February								

* updated fall 2014

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enriched Se *(radio-chemical purity)*

element	m.u.	Isotope (res.)	not distilled	distilled		
			Conv#28	Conv#30	Conv#31	Conv#36
Na	ppm	Na23(LR)	1860	<1	<1	3.3
Mg	ppb	Mg24(LR)	<1000	<100	90	<90
S	ppm	S32(MR)	1140	130	185	180
V	ppb	V51(MR)	<20	<40	<90	<90
Cr	ppb	Cr52(MR)	100	<100	<20	<20
Mn	ppb	Mn55(LR)	<100	<10	<20	<20
Fe	ppb	Fe56(MR)	300	110	<500	<500
Ni	ppb	Ni60(MR)	400	64	<100	<100
Cu	ppb	Cu63(LR)	<400	<10	27	18
Th	ppb	Th232(LR)	<0.4	<0.1	<0.1	<0.1
U	ppb	U238(LR)	<0.2	<0.1	<0.2	<0.2

HPGe test of distilled material is on the way

isotope	Selenium			Zinc
	natural	enriched	enriched and distilled	natural
^{238}U / ^{226}Ra	<1.7	<0.41	<0.3	<0.066
^{238}U / ^{234}Th	<17	<27	<6.9	<6.2
^{232}Th / ^{228}Th	1.7±0.3	1.4 ± 0.2	<0.27	<0.036
^{232}Th / ^{228}Ra	<0.7	<0.37	<0.082	<0.095
^{40}K	4 ± 2	3 ± 1	< 1.1	< 0.38
^{60}Co	<0.3	<0.17	<0.08	<0.036
^{235}U	<0.7	<0.30	<0.17	<0.09
^{137}Cs	<0.14	<0.076	<0.06	<0.033

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ZnSe synthesis

- commissioned at ISMA Kharkov, Ukraine
- improved components of the setup were made at LNGS and sent to ISMA
- synthesis of natural ZnSe is on the way and will be used for crystal growth tests
- the process is proven to be not harmful for the radio-purity of the resulting ZnSe

elem.	Isotope (resolution*)	m.u.	ZnSe synthesis #-1	#-2	#-3	#-4	#-5	#-6	thermal treatment
V	V51(MR)	ppb	<9	<11	<13	<11	<11	<8	<13
Cr	Cr52(MR)	ppb	<77	<11	<13	<15	<11	12	<105
Mn	Mn55(LR)	ppb	<19	<71	138	<65	<65	45	<26
Fe	Fe56(MR)	ppb	<385	<47	<55	200	<43	105	527
Ni	Ni58(MR)	ppb	115	95	55	130	32	120	184
Cu	Cu63(LR)	ppb	288	143	499	260	173	300	<131
Th	Th232(LR)	ppb	<1.0	<1.2	<1.4	<0.4	<0.4	<0.3	<0.5
U	U238(LR)	ppb	<0.4	<1.2	<1.4	<0.4	<0.4	0.6	<0.5

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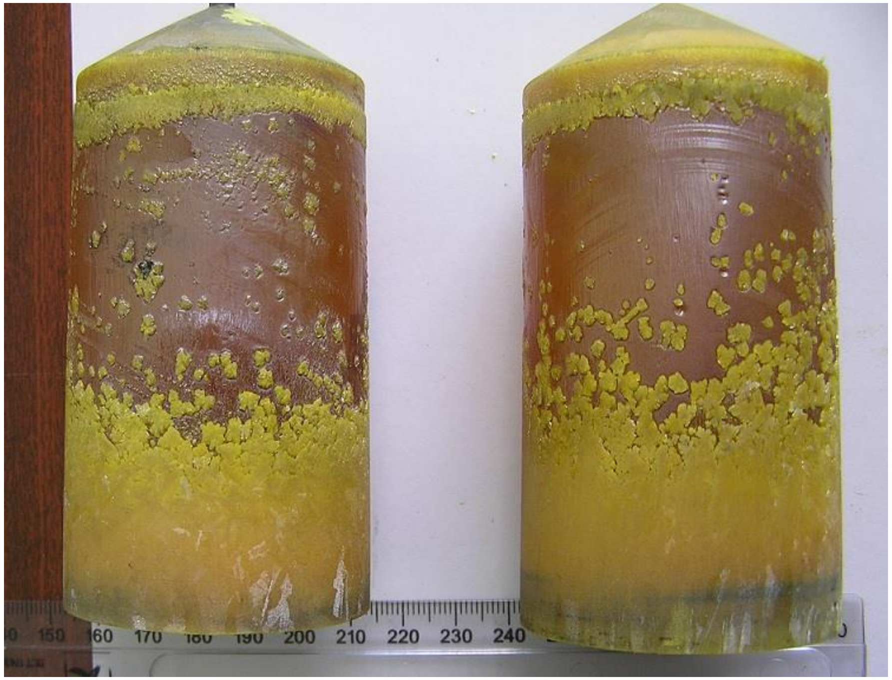
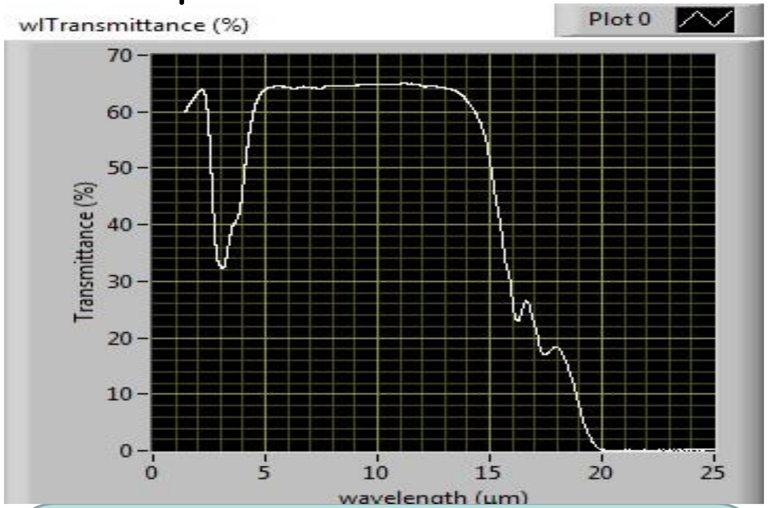
ZnSe crystal growth

- several growth test were made at **ISMA** and **ISC** in Kharkov, Ukraine in order to find the best conditions for the reliable production of crystals having standard dimensions: Ø45, H50
- issues:
  - reduced material loss
  - crucible material and geometry
  - mechanical processing of as-grown ingot



work in course for the definition of acceptance conditions



used to estimate the Fe presence in ZnSe crystals (optical absorption band at ~2.5nm). Fe^{2+} is usually paramagnetic though it may happen to have all electrons paired i.e. total spin 0; Fe^{3+} is definitely paramagnetic (1 electron) but it's quite unlikely to find Fe^{3+} in ZnSe

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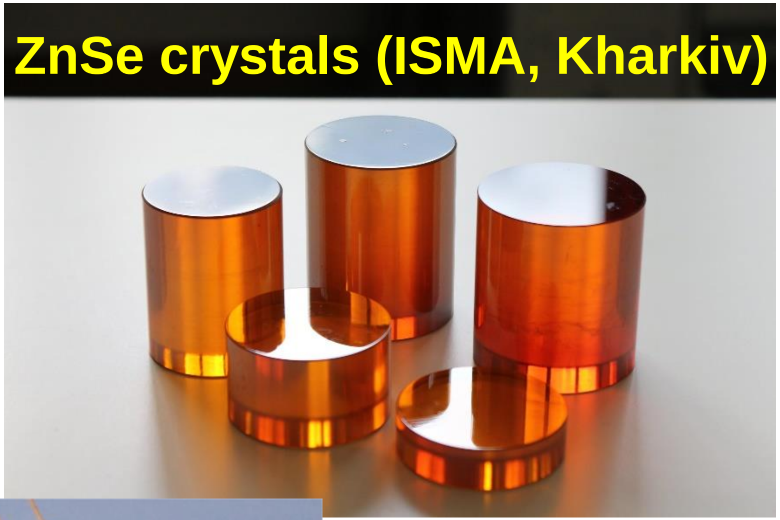
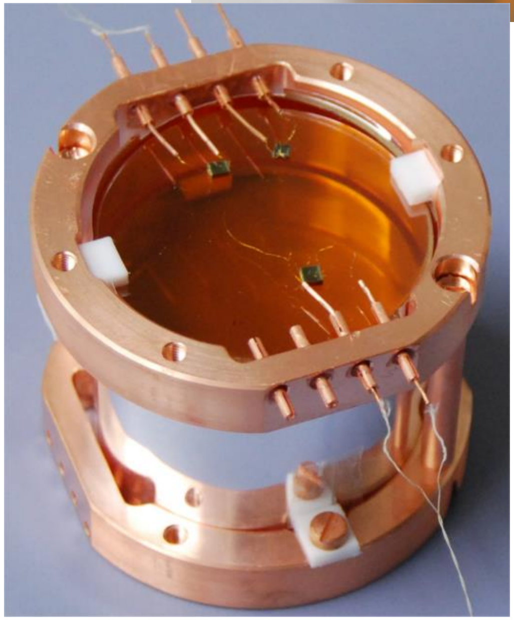
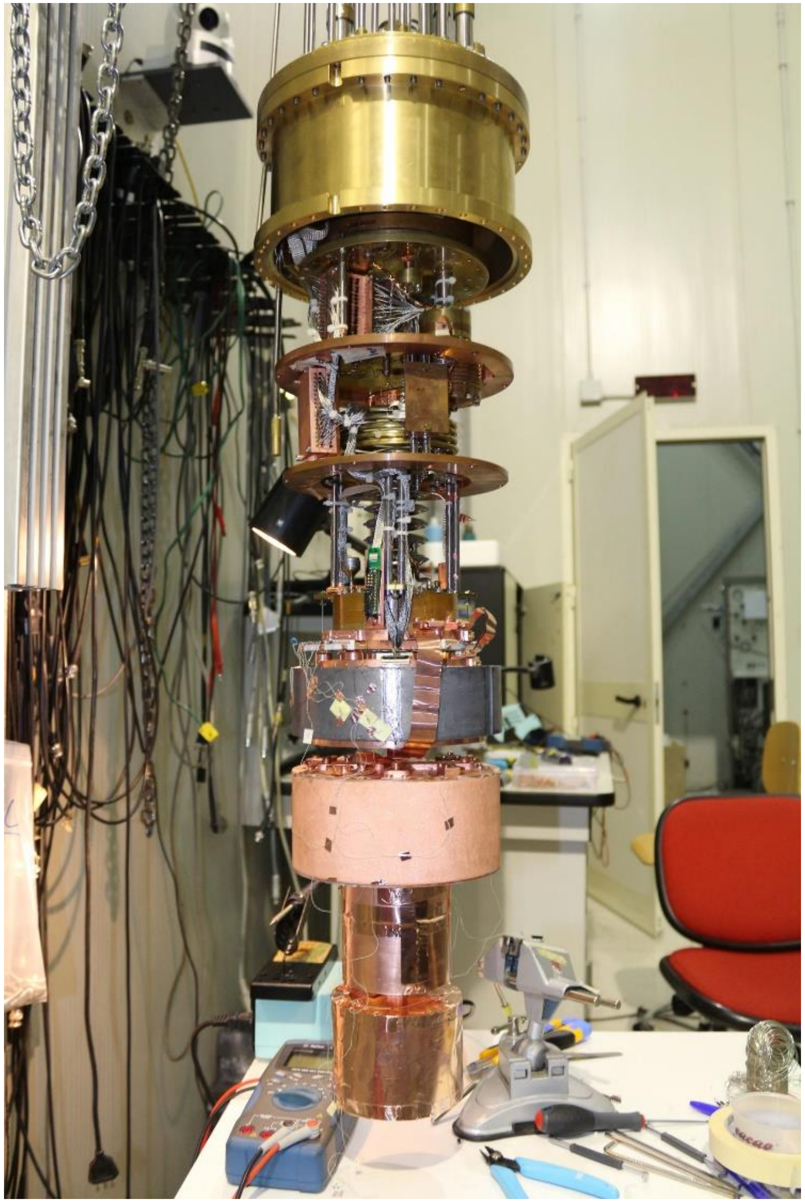
ZnSe crystal testing

Cryogenic tests at LNGS







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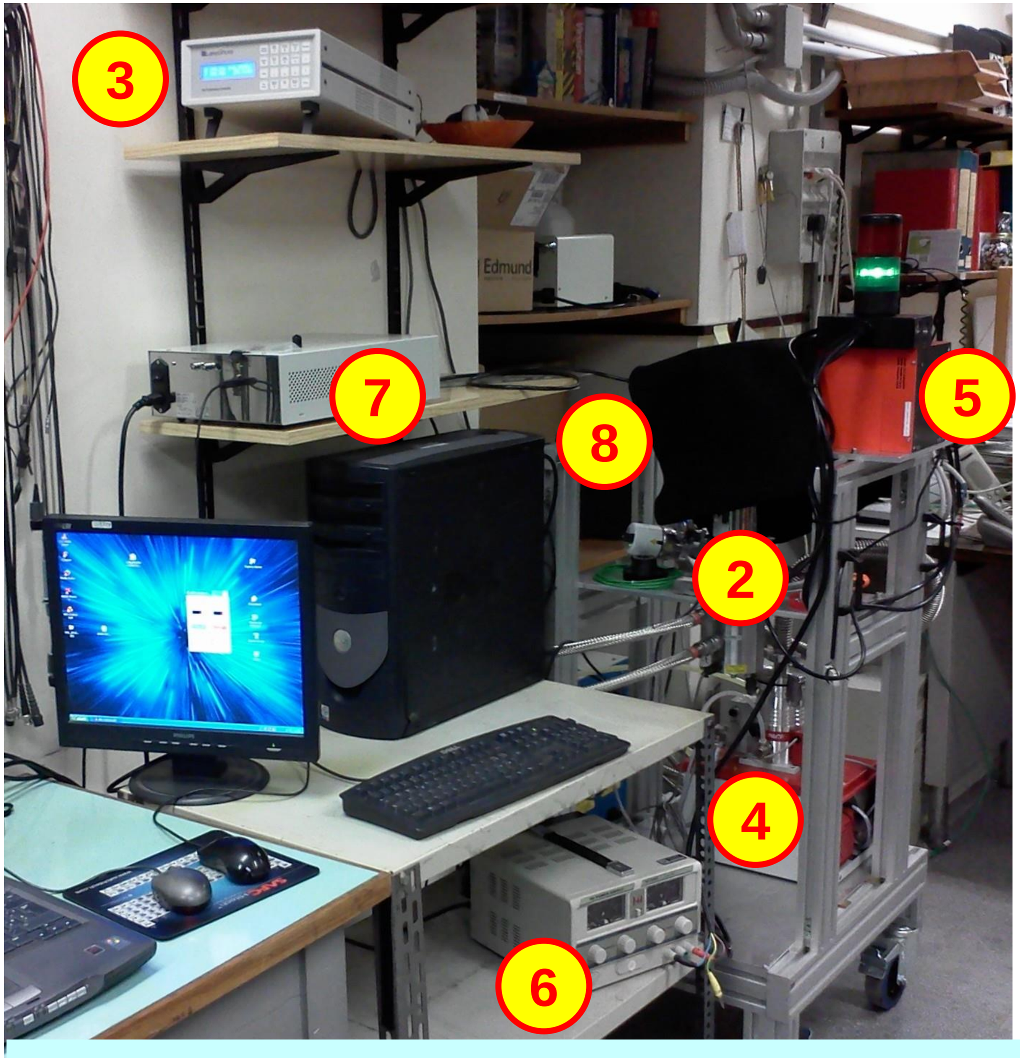
ZnSe R&D for certification

Optical measurements setup in Rome (temperature range: 10 - 300 K)



1) refrigerator: Sumitomo Helium Compressor HC-4E1
2) 10K Closed Cycle Refrigerator: Janis Research 02_CCS-100-202
3) temperature controller: Lake Shore Cryotronics, Inc. Model 325
4) vacuum pump: Pfeiffer HiCube80_Eco
5) X-ray source: Hamamatsu Microfocus 100kV L10101
6) X-ray power supply: 24V cc
7) light detector&analysis: Hamamatsu PMA-12
8) light guide: Hamamatsu quartz optic fiber

related equipment:
- oscilloscope LeCroy WR610Zi
-photoluminescence excitation source: Hamamatsu Compact UV-VIS S2D2 Fiber Light Source
- photomultipliers
- mini-monochromator
- optical fibers
- PMA-12 accessories for transmission-reflection measurements

basic equipment aimed at studying crystals and other materials at temperatures down to 10K as preliminary test for cryogenic application

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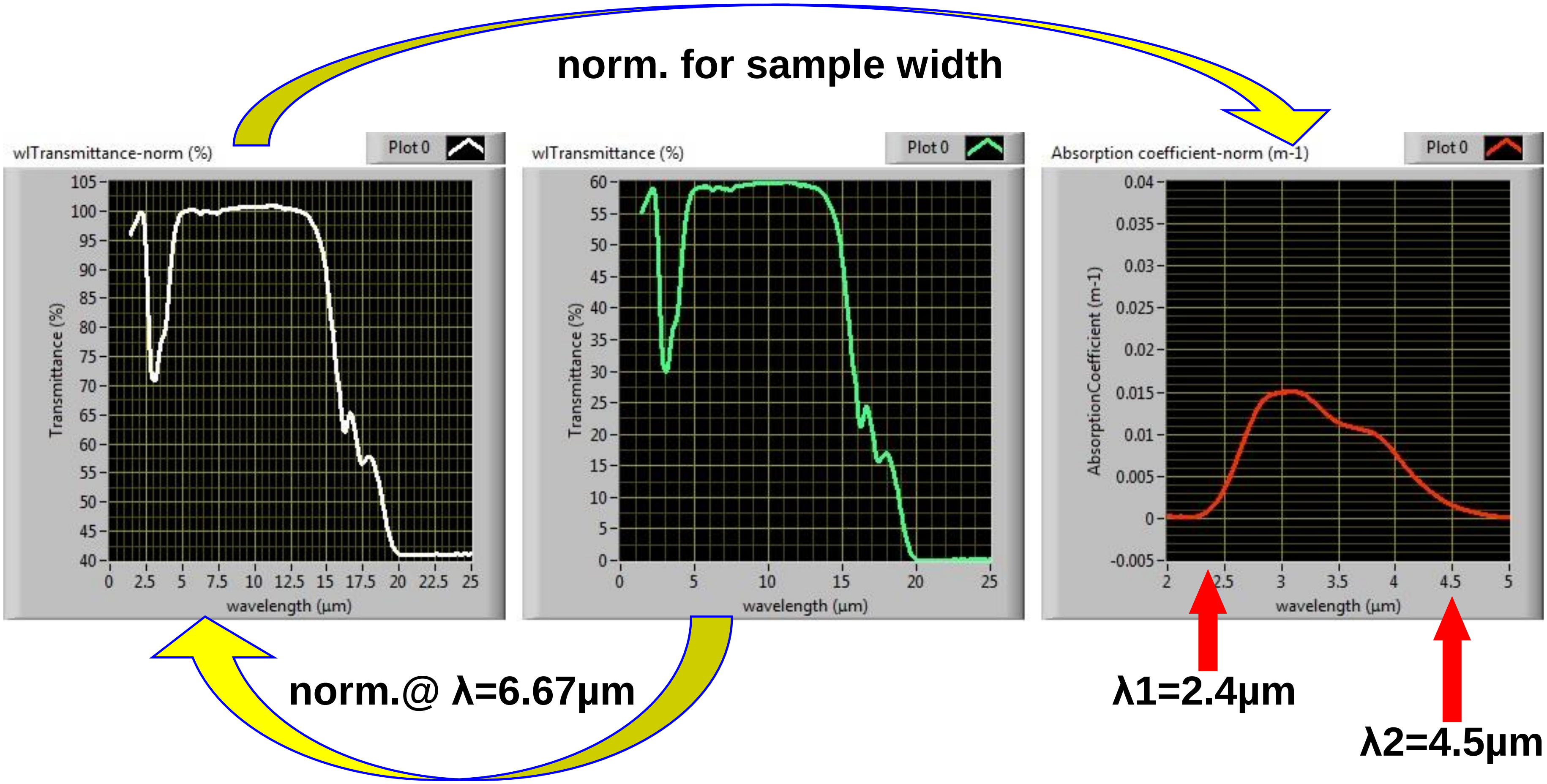
IR transmission

tests made at:
ENEA Casaccia (Dr. Baccaro)
ISMA Kharkiv (Dr. Galkin)

$$\alpha = -\frac{1}{d} \cdot \ln T$$



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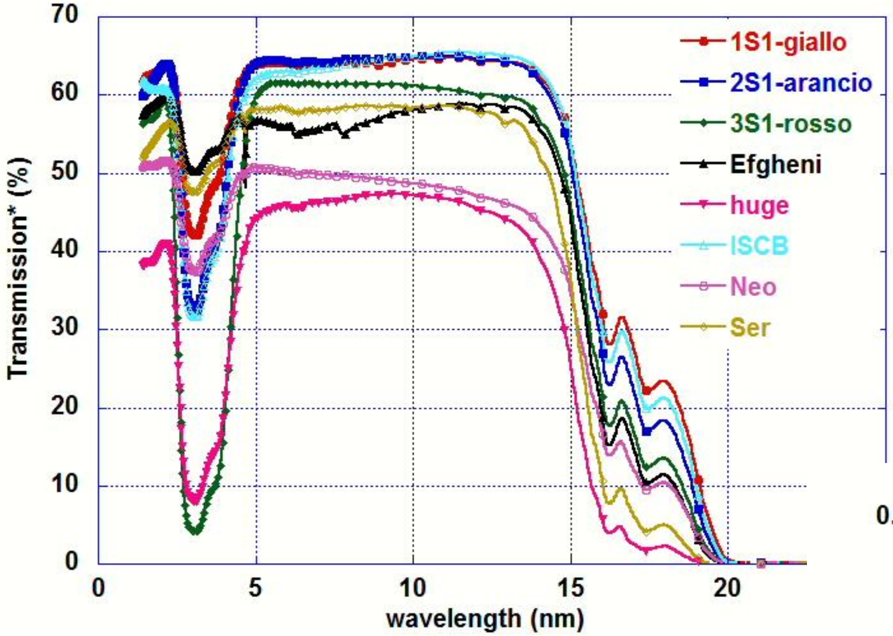
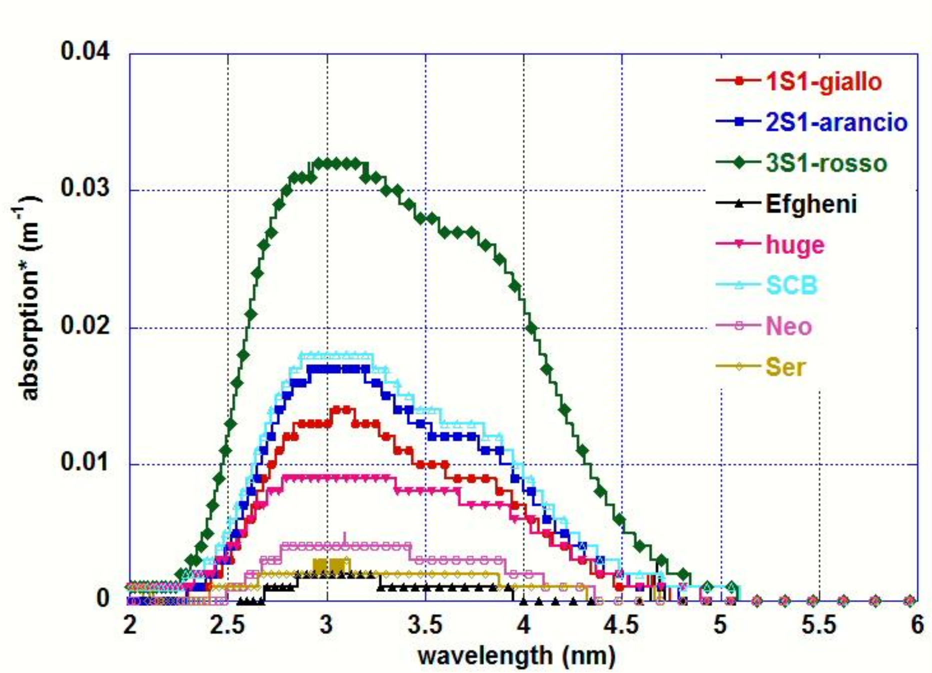
correlation search





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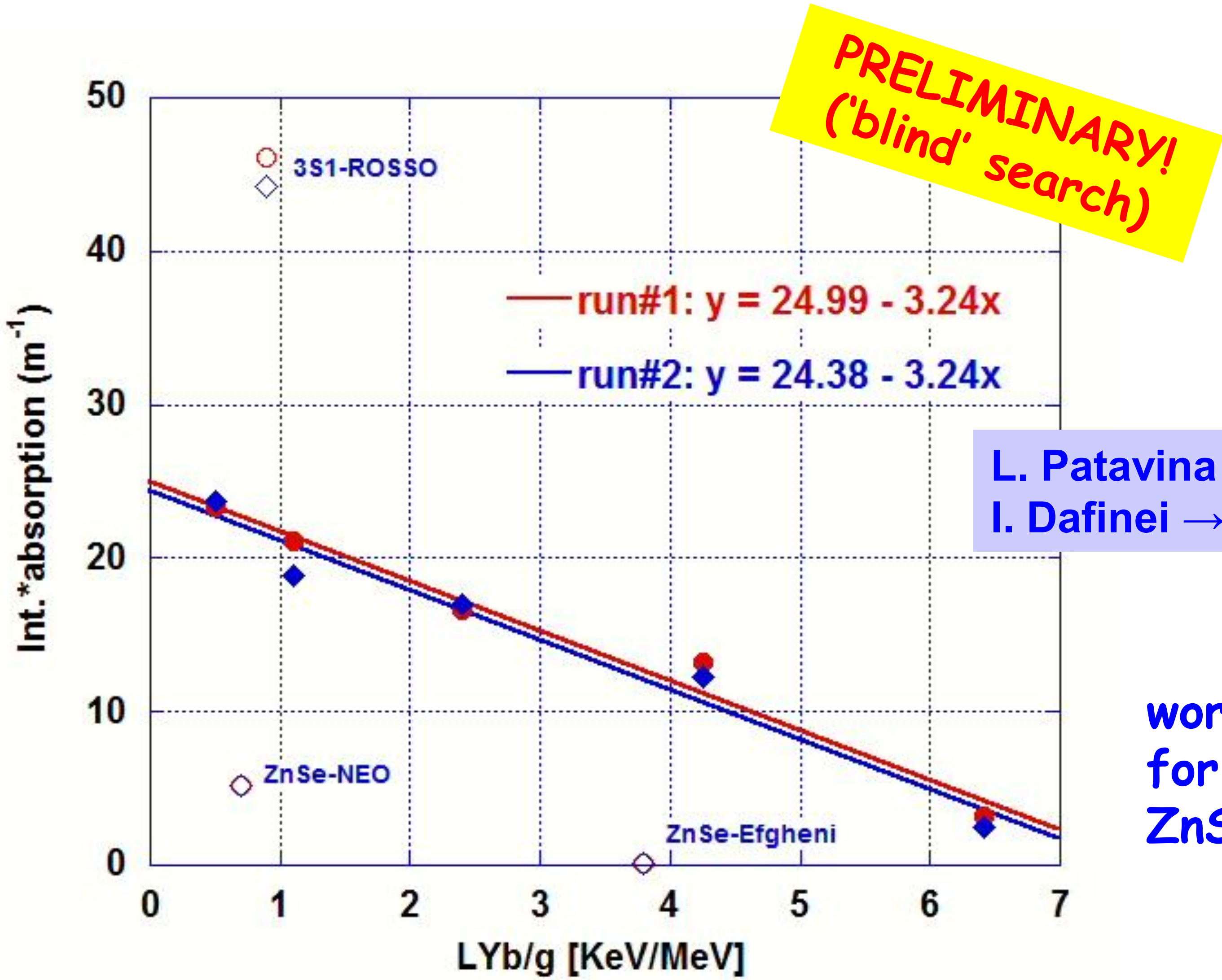
correlation search



L. Patavina → cryo parameters
I. Dafinei → optical parameters

work is on the way for another set of ZnSe crystals

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certification issues

work in progress at ISMA and INFN

the acceptance of the enriched crystal ingots will be based on the:

- knowledge that the crystals were grown from the raw material controlled, certified and delivered by INFN;
- certified synthesis of ZnSe using quartz ampoules of high purity, delivered by INFN and operated in clean conditions in the presence of INFN personnel;
- growth of ZnSe crystals in certified ultrapure crucibles (possibly delivered by INFN) to be filled with the growth charge in clean conditions in the presence of INFN personnel;
- visual inspection of the "as grown" crystal by polishing two lateral strips diametrically opposite along the growth axis of the ingot
- IR spectra along the "as grown" crystal ingot*
- cryogenic tests performed on 2-3 crystals previously produced in identical conditions from natural raw material delivered by INFN: natural (non enriched) Se and Zn

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certification issues

work in progress at INFN

- The as grown ingots of ZnSe produced from enriched material will (possibly) be cut and polished at LNGS
  - cutting machine: received, to be mounted and commissioned
  - polishing tools: purchase orders made, to be delivered soon
- It will be INFN responsibility to:
  - keep the trace of all materials used for the crystal growth in the preliminary phase (using natural elements Zn and Se);
  - ensure the same conditions along the whole production process from ZnSe synthesis to the crystal growth (as a guarantee for the reproducibility of physical properties of crystals produced);
  - recover the possible ZnSe material loss during the growth process (evaporated)
  - recover the material discarded during the mechanical processing of the crystal ingots

concluding remarks

- the definition of certification protocols for the production of 0vDBD crystals is quite difficult
- the intermediary steps of crystal production are not a (big) problem
- the certification of the final product (scintillating crystal with a good bolometric performance) is still to be defined

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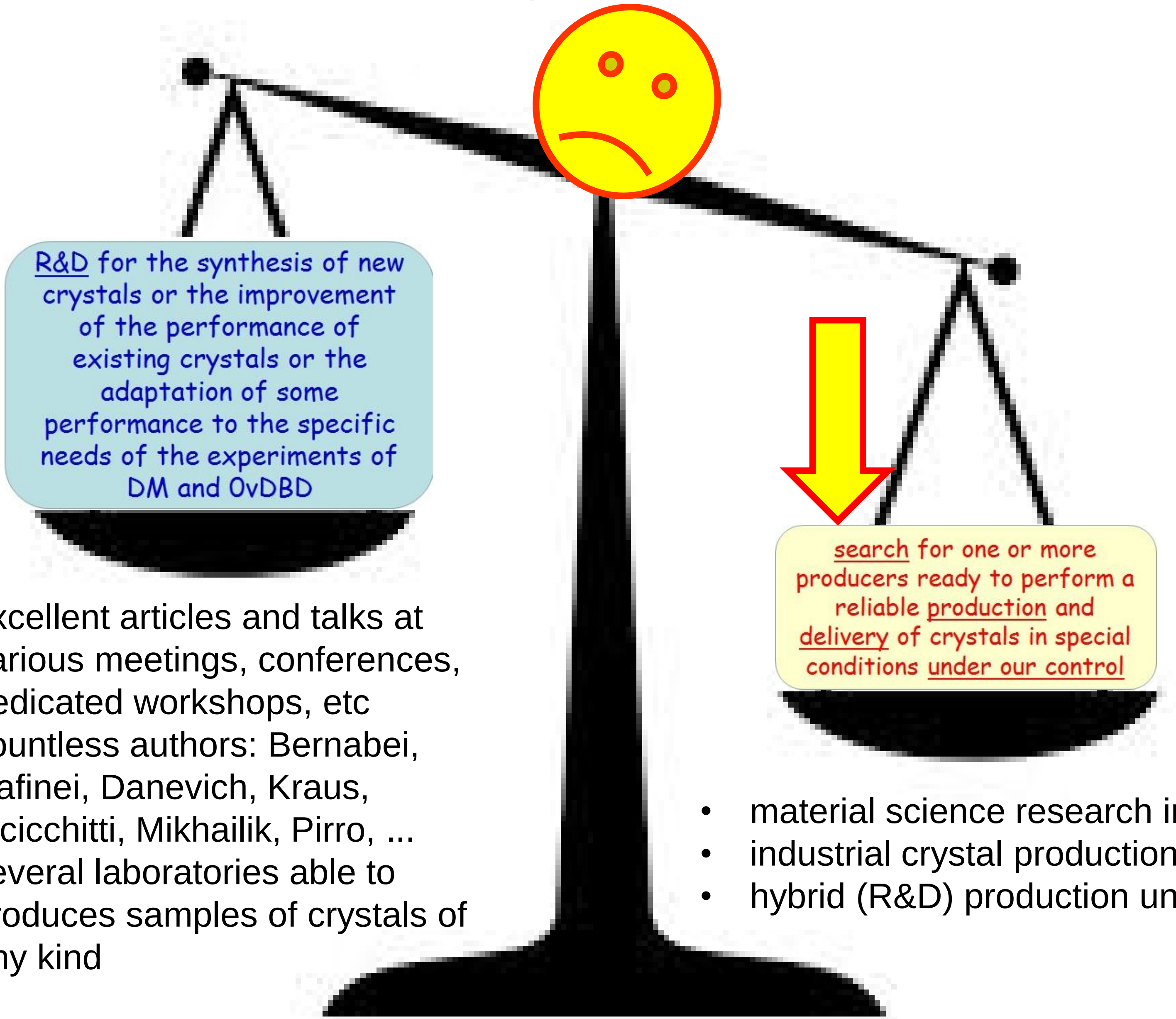
concluding remarks



- excellent articles and talks at various meetings, conferences, dedicated workshops, etc
- countless authors: Bernabei, Dafinei, Danevich, Kraus, Incicchitti, Mikhailik, Pirro, ...
- several laboratories able to produces samples of crystals of any kind

- material science research institutes: ?
- industrial crystal production units: ?
- hybrid (R&D) production units: ?

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