

Production of low radioactivity crystals for CUORE

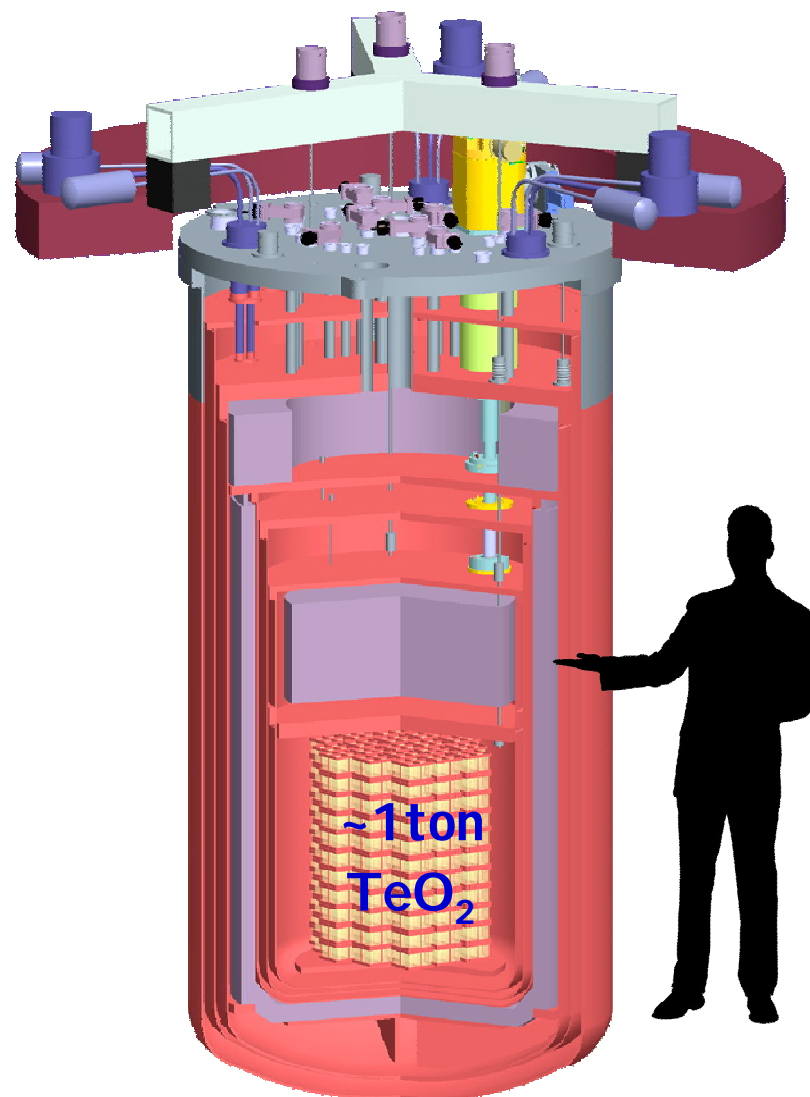
Ioan Dafinei
I.N.F.N. ITALY

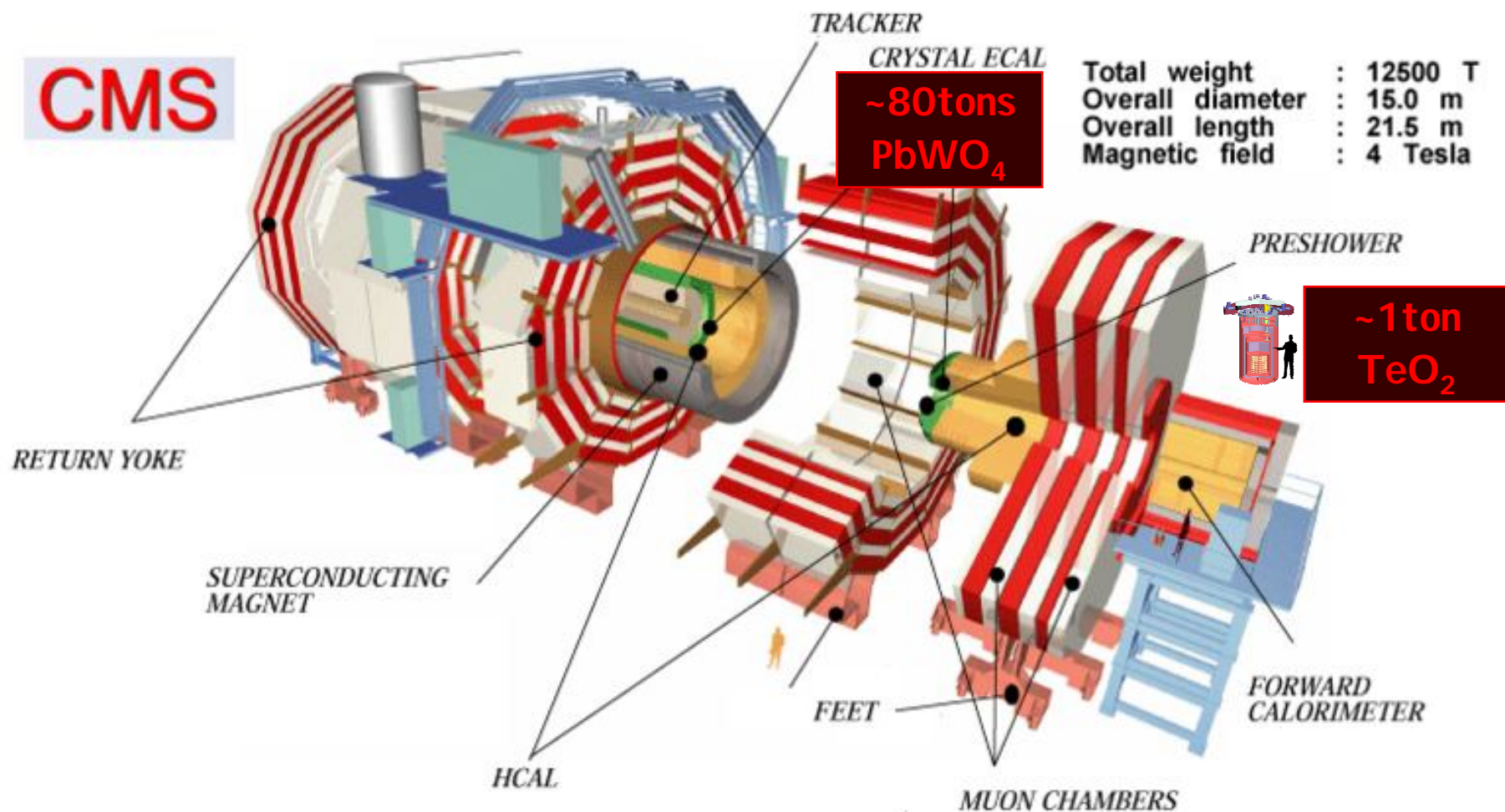
on behalf of the CUORE Collaboration

CUORE Collaboration



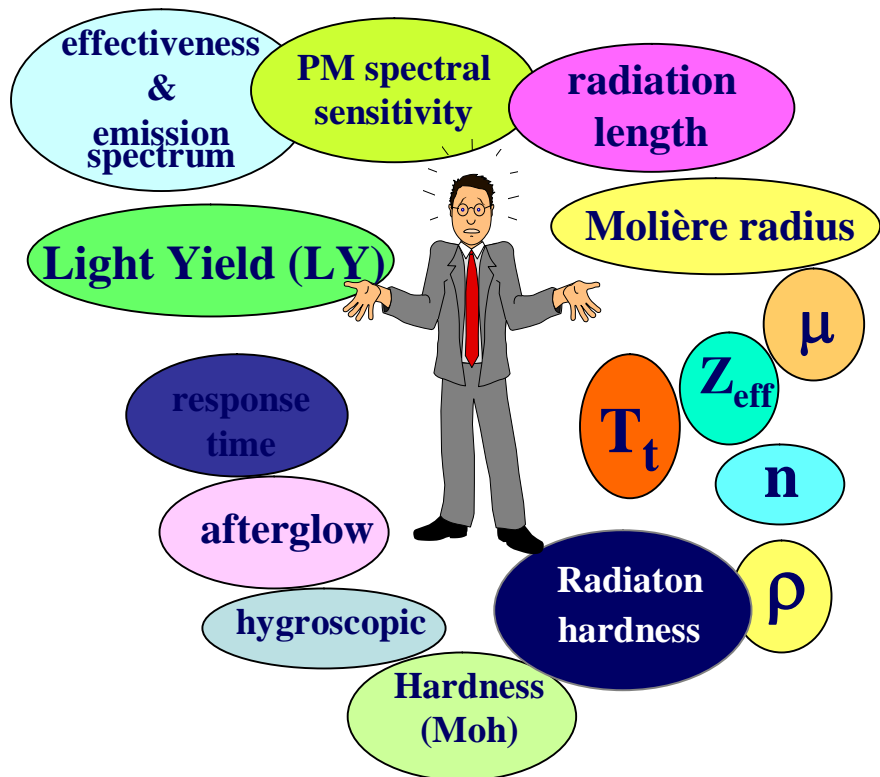
- Introduction
 - CUORE experiment, goals and consequent requirements
 - the bolometer technique
 - crystals for Rare Events Physics, technological challenge
- TeO₂ crystal
 - general properties
 - crystal growth
 - specific requirements for DBD application
- Large scale production problems
 - TeO₂ chemistry
 - crystal growth and processing
 - transport and storage
 - production management
 - certification principles and results
- Conclusions





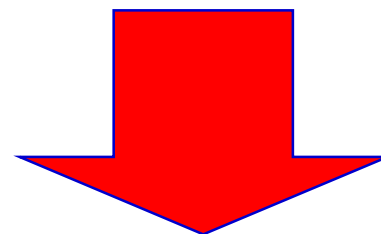
Compact Muon Solenoid

High Energy Physics (HEP) EM calorimetry



Rare Events Physics (REP)

- extremely high sensitivity
- very low background



main technological constraints

crystal perfection

radio-purity

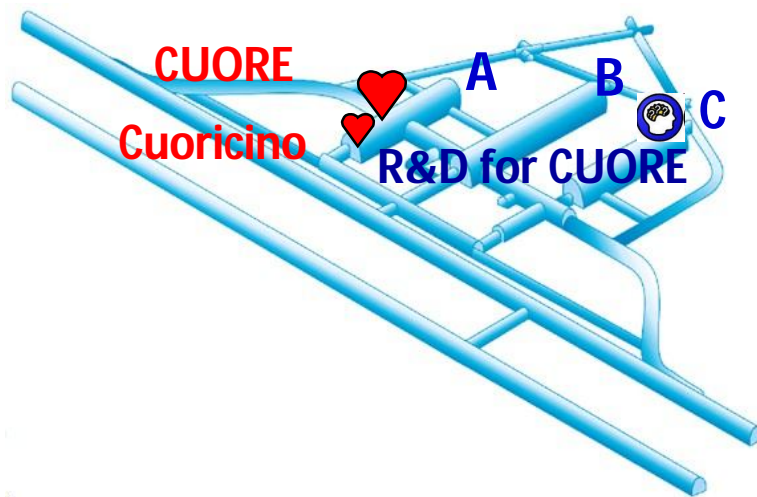
bigger challenge: quantity (in HEP) is exceeded by difficulty (in REP)

Cryogenic Underground Observatory for Rare Events



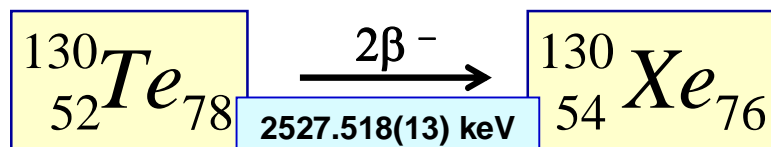
INFN, Laboratori Nazionali del Gran Sasso

(3500 m.w.e.)



- Dedicated cryostat (selected materials)
- Dilution refrigerator operated at ~ 10mK
- Dedicated shielding (including roman Pb)

• *observation and measurement of the neutrinoless double-beta decay*
 $(Z, A) \rightarrow (Z + 2, A) + 2e^-$



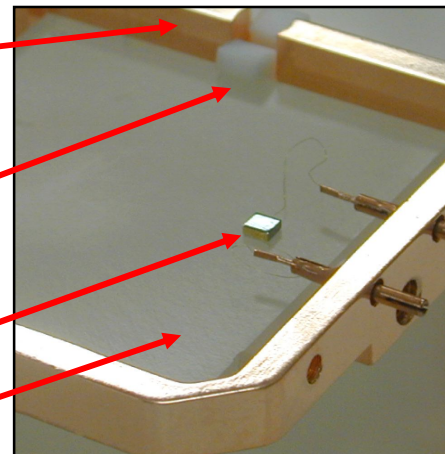
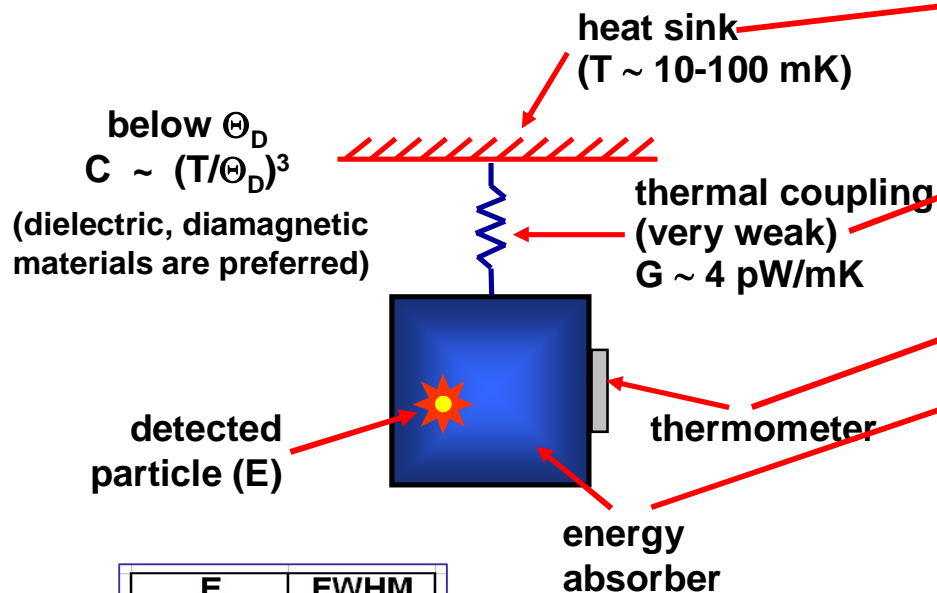
Crystals of TeO_2 are detector and source

- *observation of rare events in other energy regions*

WIMPs detection

Crystals of TeO_2 work as calorimeter

Background: 0.01 c/keV/kg/y (baseline)
 0.001 c/keV/kg/y (optimistic)
 ΔE_{FWHM} : 5 keV @ 2615 keV

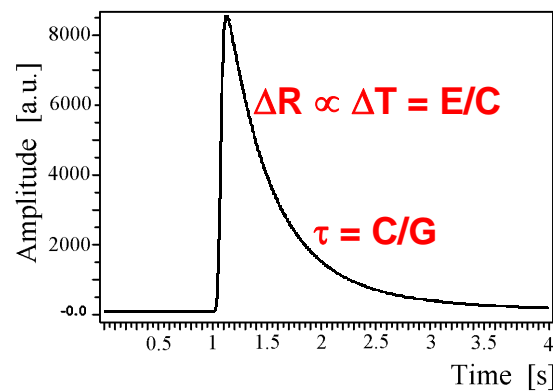
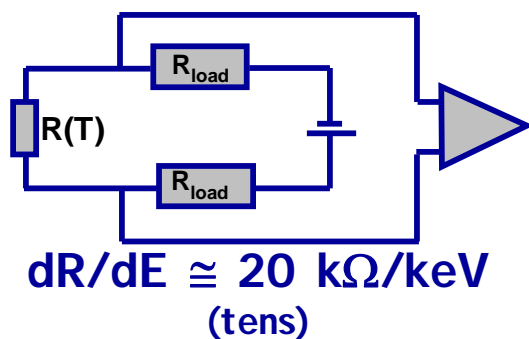


Neutron Transmutation Doped (NTD) Ge thermistors

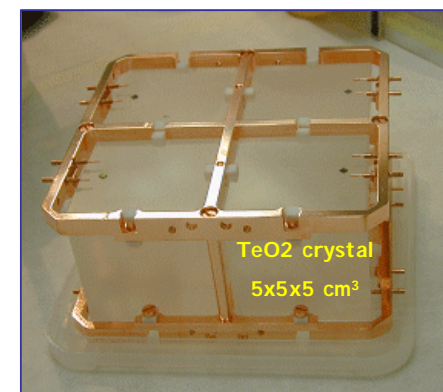
- Ge crystal exposed to neutron bombardment
- neutron capture and subsequent β and EC decay
- neutron dose controls final doping ($\sim 6 \cdot 10^{16} \text{ cm}^{-3}$)

below 10K: $\rho(T) = \rho_0 \cdot \exp\left[\left(T_0/T\right)^2\right]$
 T_0 and ρ_0 are controlled through doping

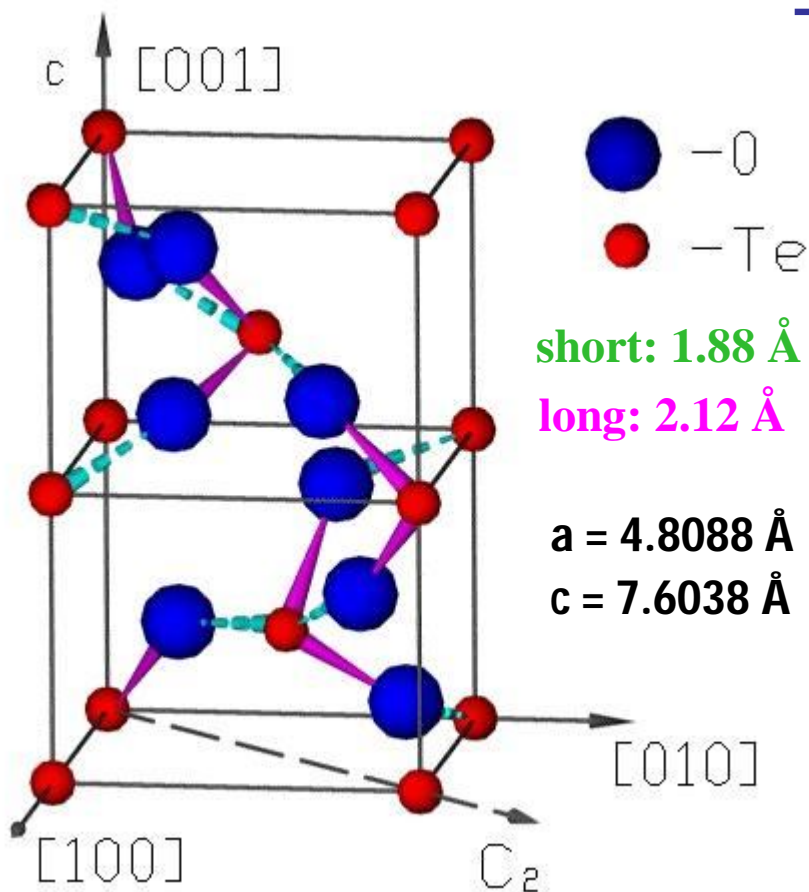
E [keV]	FWHM
46	0.8
351	1.4
911	2.1
2615	2.6
5407	3.2



CUORICINO module: 4 x 790g



Ioan Dafinei
INFN Sezione di Roma, ITALY



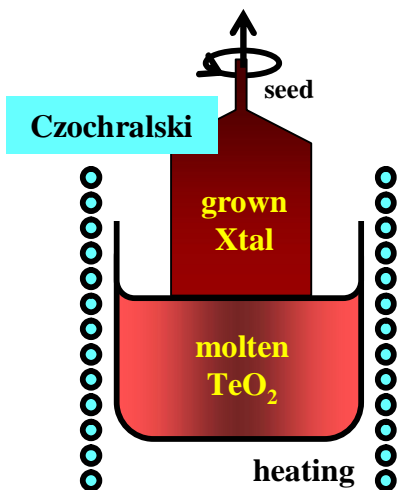
TeO₂ (paratellurite)

Characteristic	value
Chemical Formula	TeO ₂
Molecular Weight	159.61
Crystal Class	Tetragonal
Density (g/cm ³ at 20 °C)	6
Melting Point (°C)	733°C
Hardness (Mohs)	4
Solubility in water	None
Color	Clear
Transmittance Range (μm)	0.33-5.0
Refractive index (λ=500nm)	n _o =2.3194 n _e =2.4829
Thermal Expansion (1/K at 0°C)	
normal to <001>	19.5 x 10 ⁻⁶
parallel to <001>	6.10 x 10 ⁻⁶

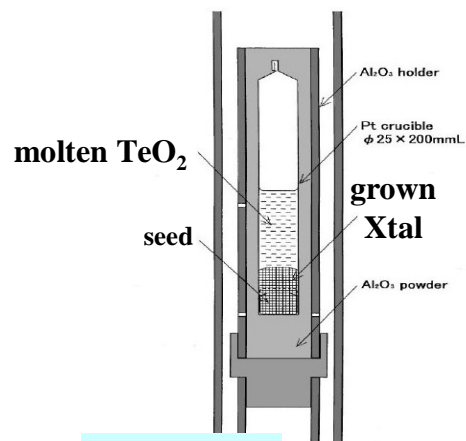
Paratellurite	Tellurite
tetragonal	orthorhombic-dipyramidal
greyish-white, opaque	white to yellow, subtranslucent to opaque
1960, Mexico	1842, Romania

- relatively low melting point
- distorted rutile (TiO₂) structure
- anisotropy of expansion coefficient

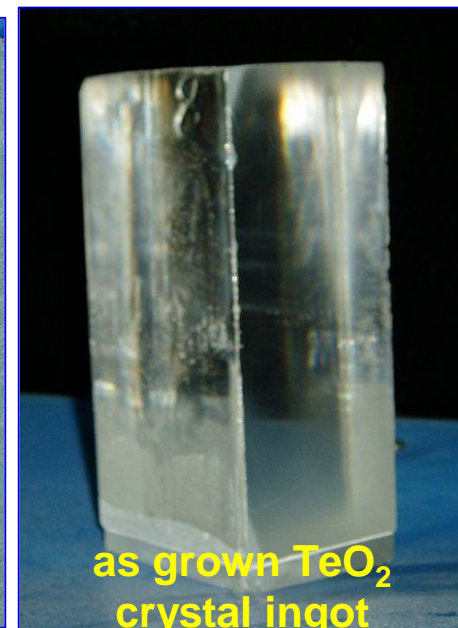
TeO₂ crystal growth



- TeO₂ crystal is particularly repellent to impurities
- most of radioactive isotopes have ionic characteristics incompatible with substitutional incorporation in TeO₂

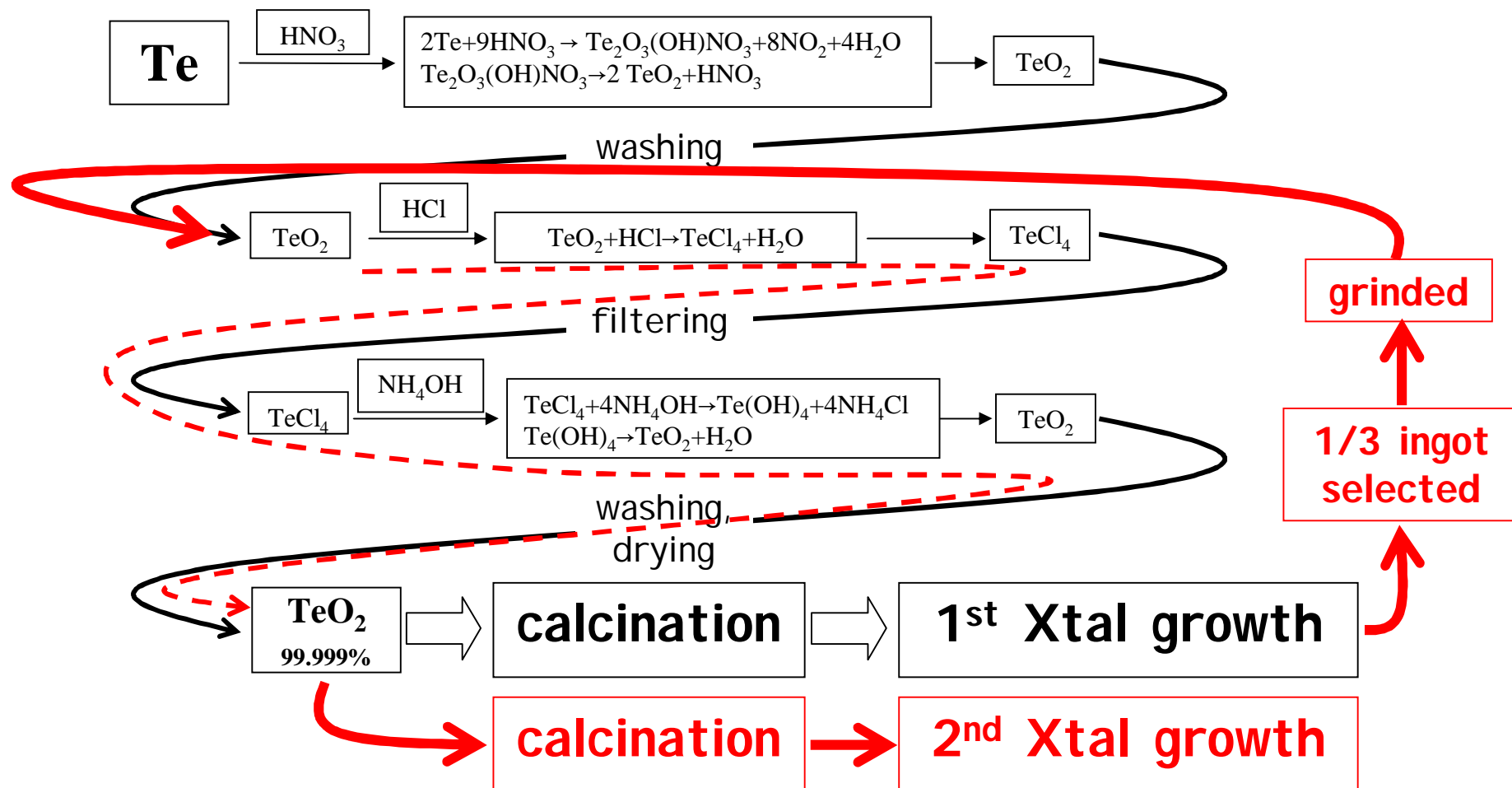


Bridgman

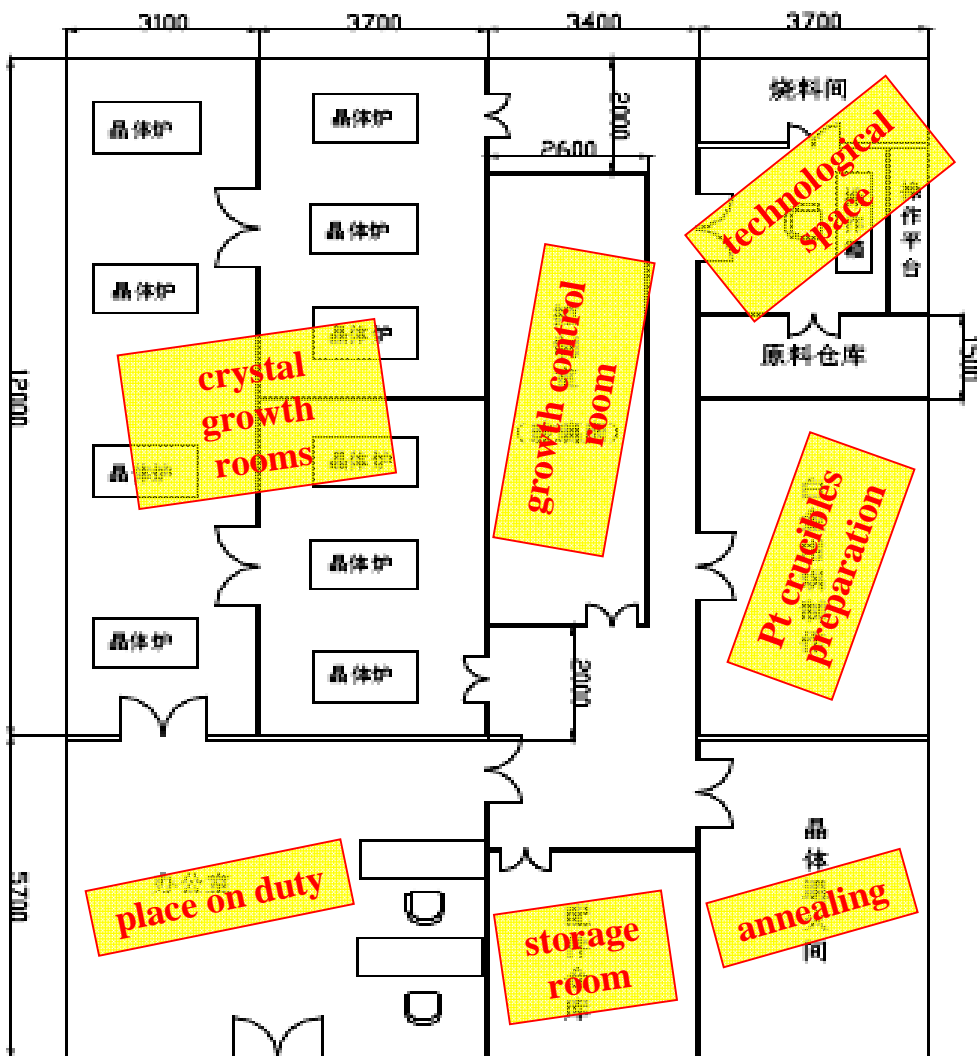


- *Bridgman grown crystals are more stressed than Czochralski ones*
- *annealing at about 550°C helps in removing the residual stresses*

refining TeO₂ @ SICCAS



TeO₂ crystal growth facility @ SICCAS



- specially dedicated area
- dedicated measures against contamination risks
- separated raw material circuit
- separated consumables and ancillaries circuit
- production flow survey by periodic certification of materials used (raw materials, consumables, ancillaries) and procedures applied

C. Arnaboldi et al, "Production of high purity TeO₂ single crystals for the study of neutrinoless double beta decay", *Journal of Crystal Growth* (Article in Press), doi:10.1016/j.jcrysgr.2010.06.034



- CUORE dedicated mechanical processing area
 - new cutting machines (inner and outer cut)
 - new grinding machine
 - new lapping bench



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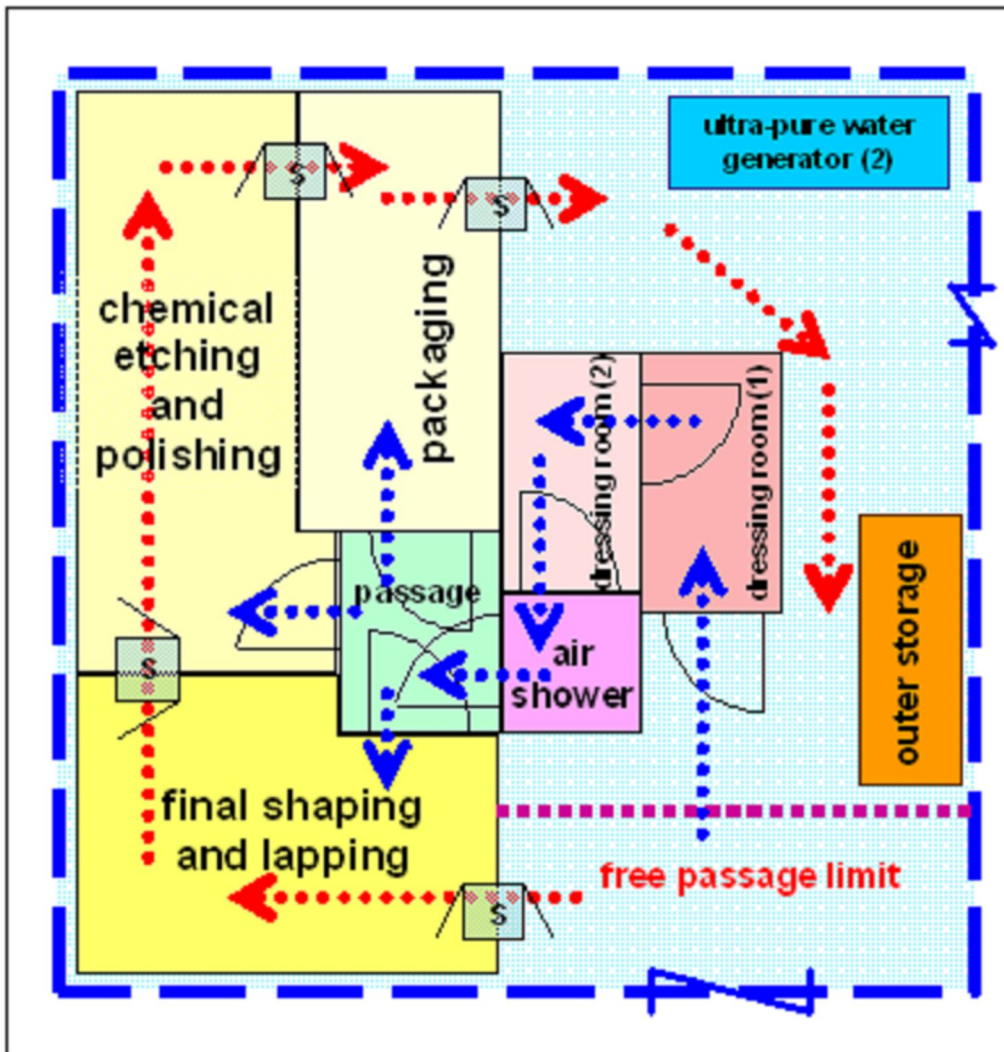


SICCAS/INFN Clean Room for TeO₂ final processing at Jiading

- class 1000 qualified
- general standard respected
- easy maintenance
- ultrapure water 300 l/h

equipment:

- ultrasound washer
- microwave oven
- chemical etching bench
- polishing bench
- packaging bench



SICCAS Jiading

package



triple vacuum bags



vacuum boxes

shipment: cosmogenic contamination risk

- total exposure time to cosmic rays from TeO₂ synthesis to LNGS delivery < 6 months
- sea transport from China to Italy, in order to minimize cosmic rays exposure

storage: radon contamination risk



visual inspection at delivery



LNGS permanent storage area



technical specifications

raw materials and reactants

Te metal contamination

$$^{238}\text{U} < 2 \cdot 10^{-10} \text{ g/g}$$

$$^{232}\text{Th} < 2 \cdot 10^{-10} \text{ g/g}$$

$$^{210}\text{Pb} < 10^{-4} \text{ Bq/kg}$$

$$^{40}\text{K} < 10^{-3} \text{ Bq/kg}$$

$$^{60}\text{Co} < 10^{-5} \text{ Bq/kg}$$

acids contamination

$$^{238}\text{U} < 5 \cdot 10^{-12} \text{ g/g}$$

$$^{232}\text{Th} < 5 \cdot 10^{-12} \text{ g/g}$$

water contamination

$$^{238}\text{U} < 4 \cdot 10^{-12} \text{ g/g}$$

$$^{232}\text{Th} < 4 \cdot 10^{-12} \text{ g/g}$$

contamination of other liquids

$$^{238}\text{U} < 5 \cdot 10^{-12} \text{ g/g}$$

$$^{232}\text{Th} < 5 \cdot 10^{-12} \text{ g/g}$$

intermediary products

TeO₂ powder (after densification by calcination)

$$^{238}\text{U} < 2 \cdot 10^{-10} \text{ g/g}$$

$$^{232}\text{Th} < 2 \cdot 10^{-10} \text{ g/g}$$

$$^{210}\text{Pb} < 10^{-4} \text{ Bq/kg}$$

$$^{40}\text{K} < 10^{-3} \text{ Bq/kg}$$

$$^{60}\text{Co} < 4 \cdot 10^{-5} \text{ Bq/kg}$$

Pt contamination in as grown crystals

$$\text{Pt (element)} < 10^{-10} \text{ g/g}$$

$$^{190}\text{Pt (isotope)} < 3 \cdot 10^{-6} \text{ Bq/kg}$$

final product

(TeO₂ crystal ready-to-use)

$$^{238}\text{U} < 3 \cdot 10^{-13} \text{ g/g}$$

$$^{232}\text{Th} < 3 \cdot 10^{-13} \text{ g/g}$$

$$^{210}\text{Pb} < 10^{-5} \text{ Bq/kg}$$

$$^{60}\text{Co} < 10^{-6} \text{ Bq/kg}$$

final product

(bolometer)

$$\text{energy resolution} < 15 \text{ keV at } 2615 \text{ keV}$$

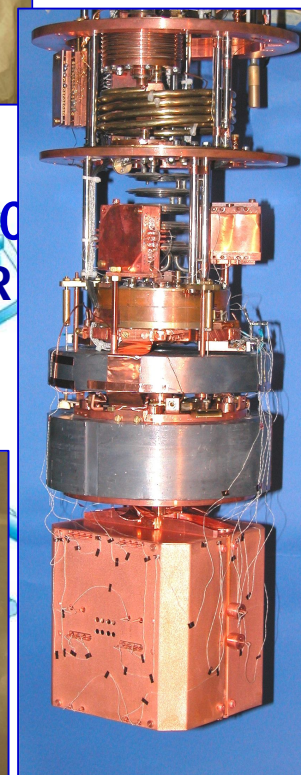
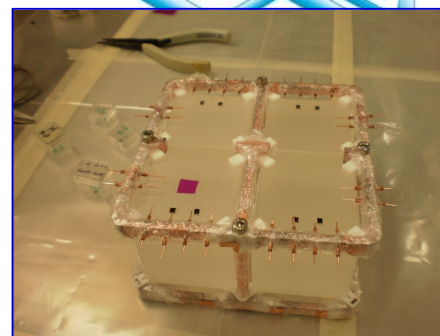
$$\text{signal intensity} > 60 \mu\text{V/MeV}$$

objectives

- bolometer performance:
 - energy resolution
 - detector response
- bulk contaminations (from alpha peaks). Able to get limits on the contamination levels in two weeks, at the 10^{-12} g/g level.
- surface contaminations (from coincident events and from distortion in the peaks). Needs more statistics than bulk contaminations.
- measurement of the background reduction factor from surface cleaning



LNGS



CCVR map

no.	Berkeley		INFN																			
	Te-01	Te-02	Te-03			Te-04				Te-05				Te-06				Te-07				
	1a	1b	1	2	3	4a	4b	5	6	7	8	9	10	11	12	13	14	15	16	17		
1	080005	080039	080070	080099	080137	080171	080203	080237	080275	080312	080349	080382	080410	080444	080479							
2	080006	080040	080071	080100	080138	080172	080204	080238	080276	080313	080350	080383	080411	080445	080480							
3	080007	080041	080072	080101	080139	080173	080206	080239	080277	080314	080351	080384	080412	080446	080481							
4	080008	080042	080073	080102	080140	080174	080208	080240	080278	080315	080352	080385	080413	080447	080482							
5	080009	080043	080074	080103	080141	080175	080209	080241	080279	080316	080353	080386	080414	080448	080483							
6	080011	080044	080075	080104	080142	080176	080210	080242	080280	080318	080354	080387	080415	080449	080484							
7	080012	080045	080076	080105	080144	080177	080211	080243	080281	080319	080355	080388	080416	080450	080485							
8	080014	080046	080077	080106	080145	080178	080212	080244	080282	080320	080356	080389	080417	080451	080487							
9	080015	080047	080078	080107	080146	080179	080213	080246	080283	080322	080358	080390	080418	080452	080488							
10	080016	080048	080079	080108	080147	080180	080214	080247	080284	080323	080359	080391	080419	080453	080489							
11	080017	080049	080080	080109	080148	080181	080215	080248	080285	080325	080360	080392	080420	080454	080490							
12	080018	080050	080081	080110	080149	080182	080216	080249	080286	080326	080361	080393	080421	080455	080491							
13	080019	080051	080082	080111	080150	080184	080217	080250	080287	080327	080362	080395	080422	080456	080492							
14	080020	080052	080083	080112	080151	080185	080218	080252	080289	080328	080363	080396	080424	080457	080493							
15	080021	080053	080084	080113	080152	080186	080219	080253	080290	080329	080364	080397	080425	080459	080494							
16	080022	080054	080085	080114	080153	080187	080220	080254	080291	080330	080365	080398	080426	080460	080495							
17	080023	080055	080087	080115	080154	080188	080221	080255	080293	080331	080366	080399	080427	080461	080496							
18	080024	080056	080088	080116	080155	080189	080222	080256	080294	080332	080367	080400	080428	080462	080497							
19	080025	080057	080089	080117	080156	080190	080223	080257	080295	080333	080368	080401	080429	080463	080498							
20	080026	080058	080090	080119	080157	080191	080224	080258	080296	080334	080369	080402	080430	080464	080499							
21	080027	080059	080091	080120	080158	080192	080226	080259	080297	080335	080370	080403	080431	080466	080500							
22	080028	080060	080092	080121	080159	080193	080227	080261	080298	080336	080371	080404	080432	080467	080501							
23	080029	080061	080094	080122	080160	080195	080228	080262	080299	080337	080372	080405	080433	080468	080502							
24	080030	080062	080095	080123	080162	080196	080229	080263	080300	080338	080373	080406	080434	080469	080503							
25	080031	080063	080096	080124	080163	080197	080230	080264	080301	080339	080374	080407	080435	080470	080504							
26	080032	080064	080097	080125	080164	080198	080231	080266	080302	080340	080375	080408	080437	080471	080505							
27	080033	080065		080126	080165	080199	080232	080267	080304	080341	080376	080409	080438	080472	080506							
28	080034	080066		080127	080166	080200	080233	080268	080306	080342	080377		080439	080473	080507							
29	080035	080067		080128	080167	080201	080234	080269	080307	080343	080378		080440	080475	080508							
30	080036	080068		080129	080168	080202	080236	080270	080308	080344	080379		080441	080476	080509							
31	080037	080069		080130	080169			080271	080309	080345	080380		080442	080477	080510							
32	080038			080131	080170			080272	080310	080346	080381		080443	080478	080511							
33				080133				080273	080311	080347												
34				080134				080274		080348												
35				080135																		
36				080136																		
CCVR	air shipment		sea shipment																			
	2/32	2/31	2/94			4/127				7/125												
	6.25%	6.45%	2.13%			3.15%				5.60%												

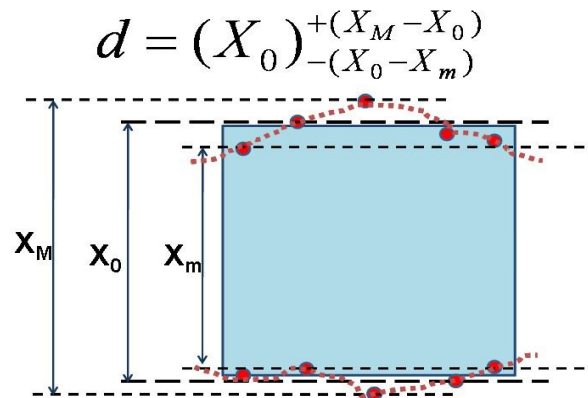
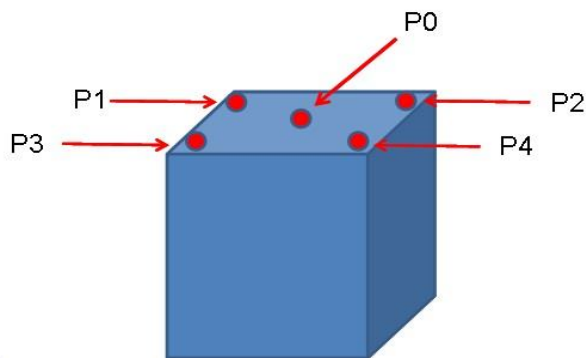
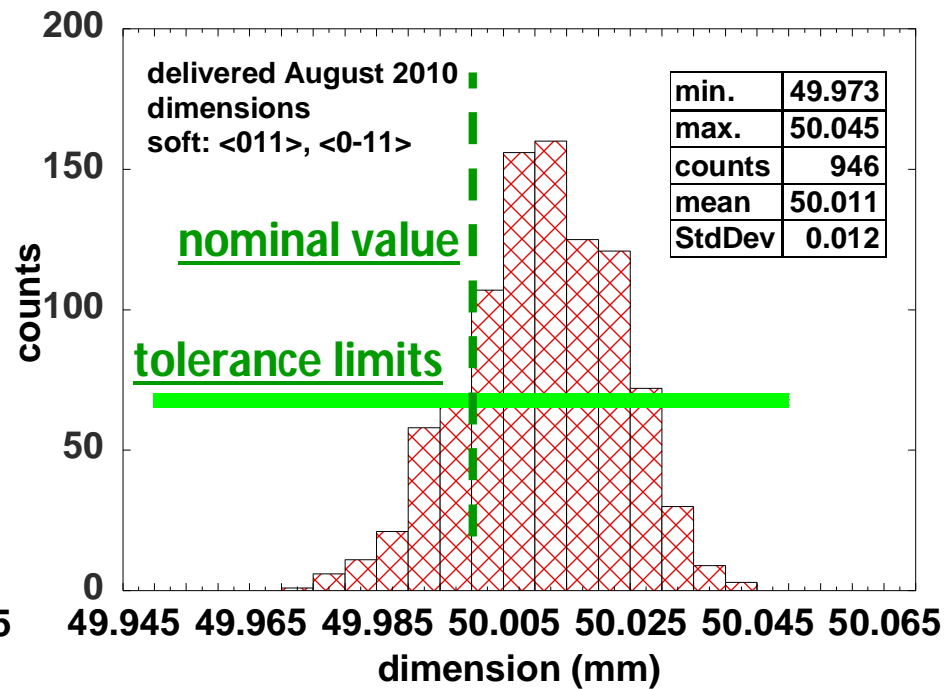
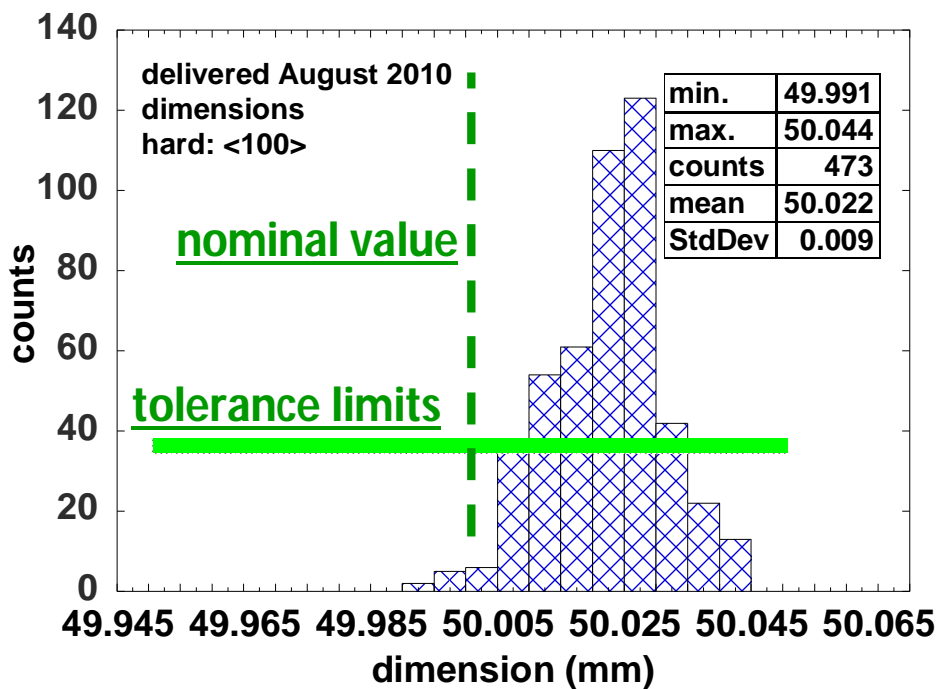
CCVR results

<http://wiki.hep.wisc.edu/cuore/HallC>

Crystal	^{238}U (g/g)	^{232}Th (g/g)	FWHM (keV) @ 1460 keV	FWHM (keV) @ 2615 keV	FWHM (keV) @ 5407 keV
007	<1.8E-14	<5.5E-14		3.05	2.78
011				3.63	3.42
039				4.22	3.95
041				4.58	4.73
076	<3.12E-13	<2.26E-13		4.40	4.70
096	<2.20E-13	<8.52E-14		4.20	3.20
180	<4.4E-13	<4.7E-14	4.1		4.7
190			3.9		4.6
229			4.3		4.1
236			6.2		7.2
313	<9.0E-14	<3.7E-13		9.90	6.90
340				6.90	6.00
354				11.90	5.40
380				9.80	9.30

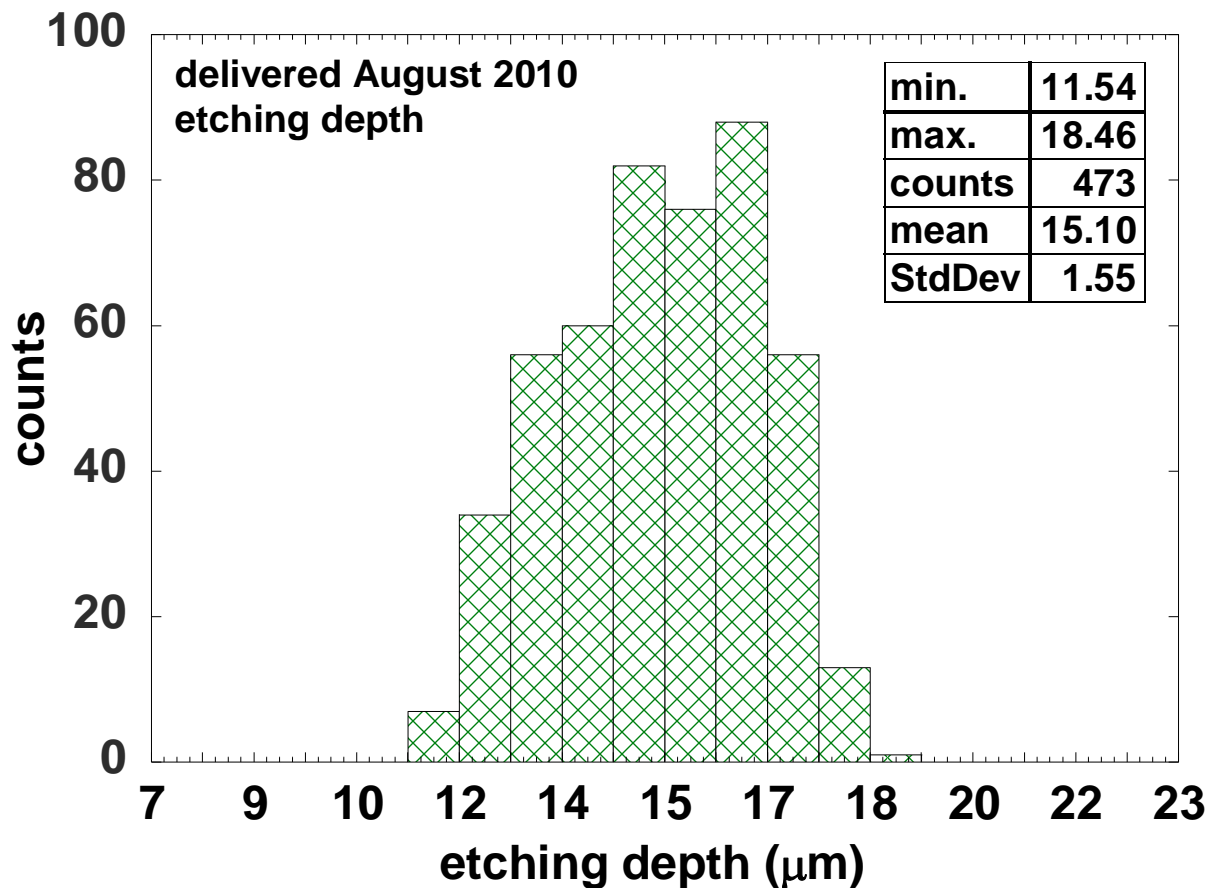
crystal dimensions

crystals (July 2010): 473



crystal surface treatment (1)

etching



atomic layers

-soft face: 1470/ μ

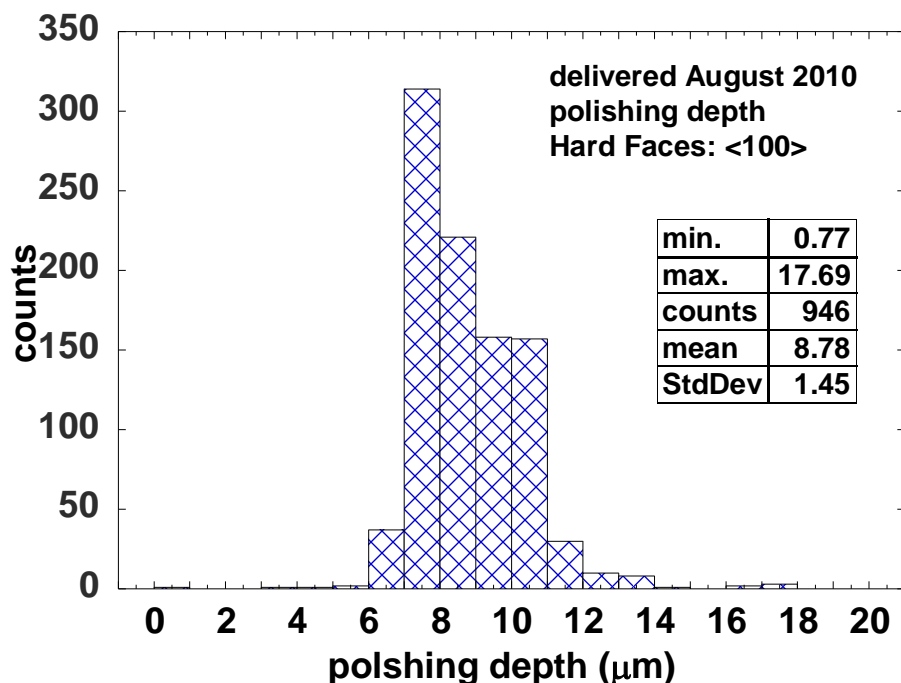
-hard face: 1320/ μ

atomic layers taken
away by etching:

-soft face: >22000 layers

-hard face: >20000 layers

crystal surface treatment (2)



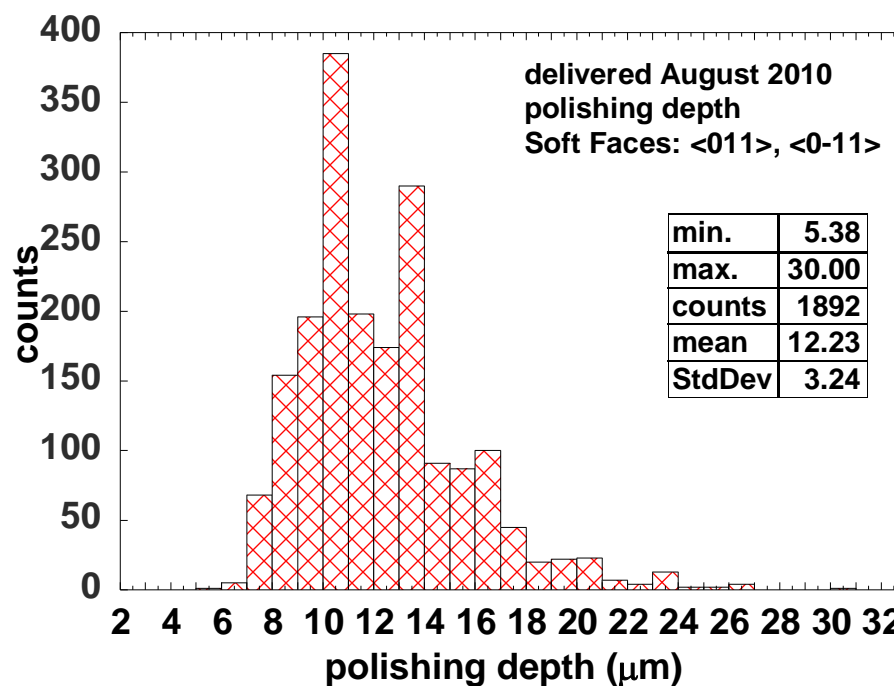
atomic layers taken away by polishing:

- hard face: ~ 11600 layers
- soft face: ~ 18000 layers

polishing

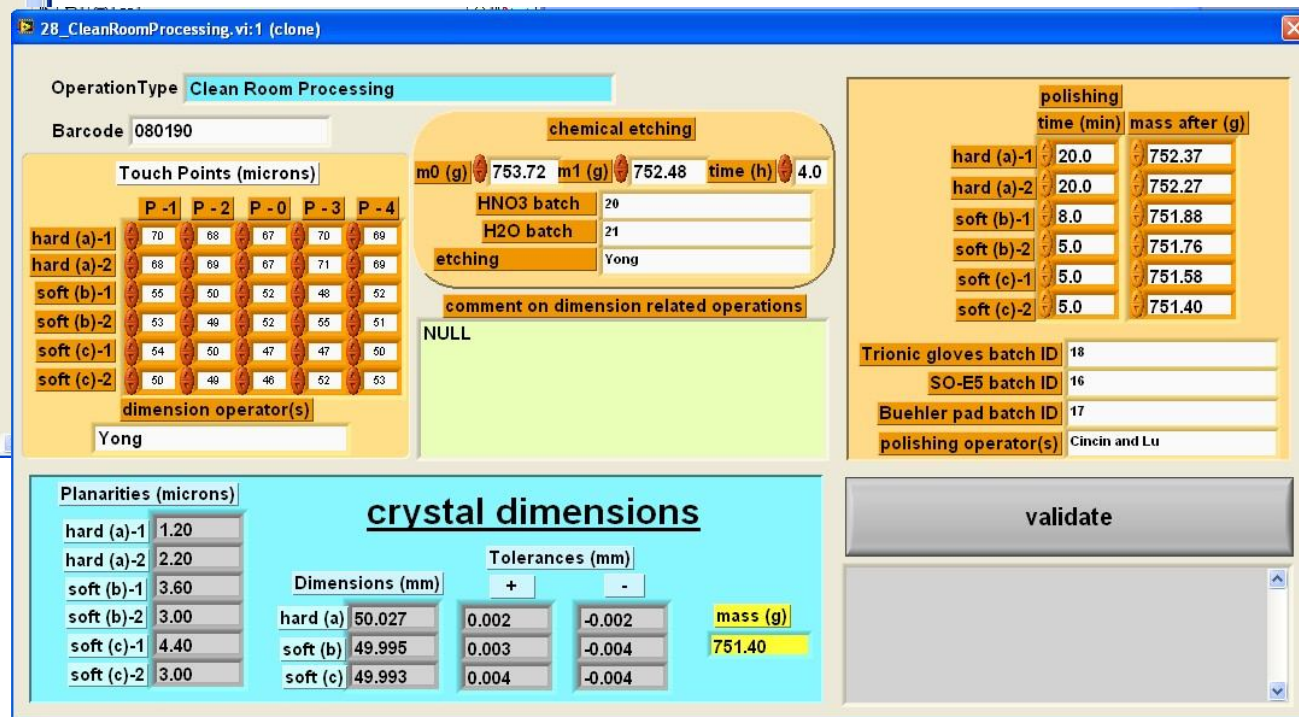
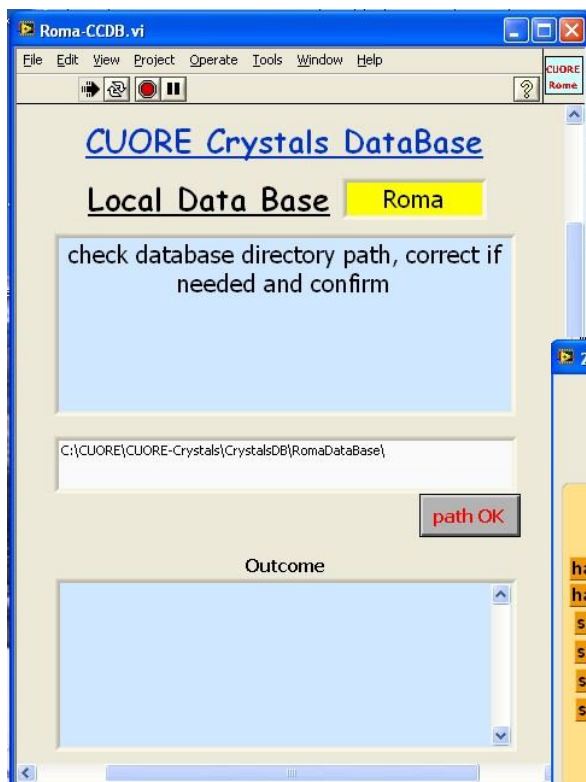
atomic layers

- soft face: 1470/ μ
- hard face: 1320/ μ

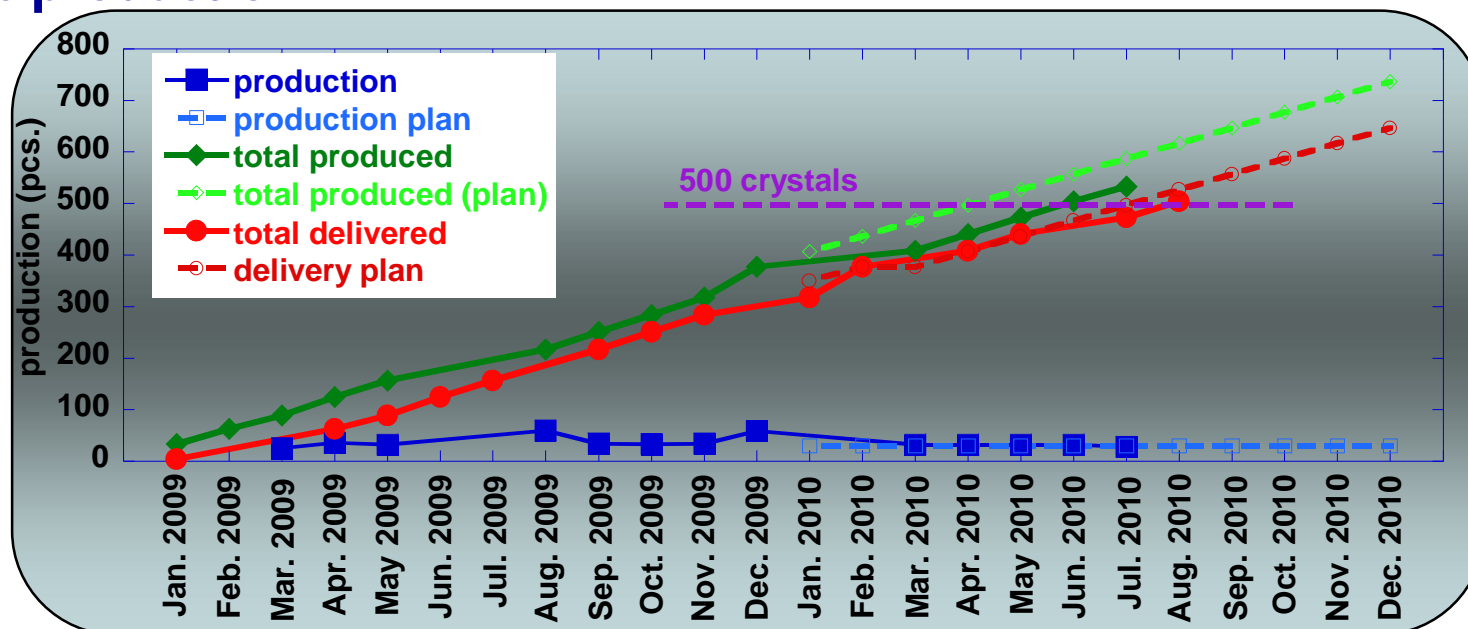


CUORE crystals production database

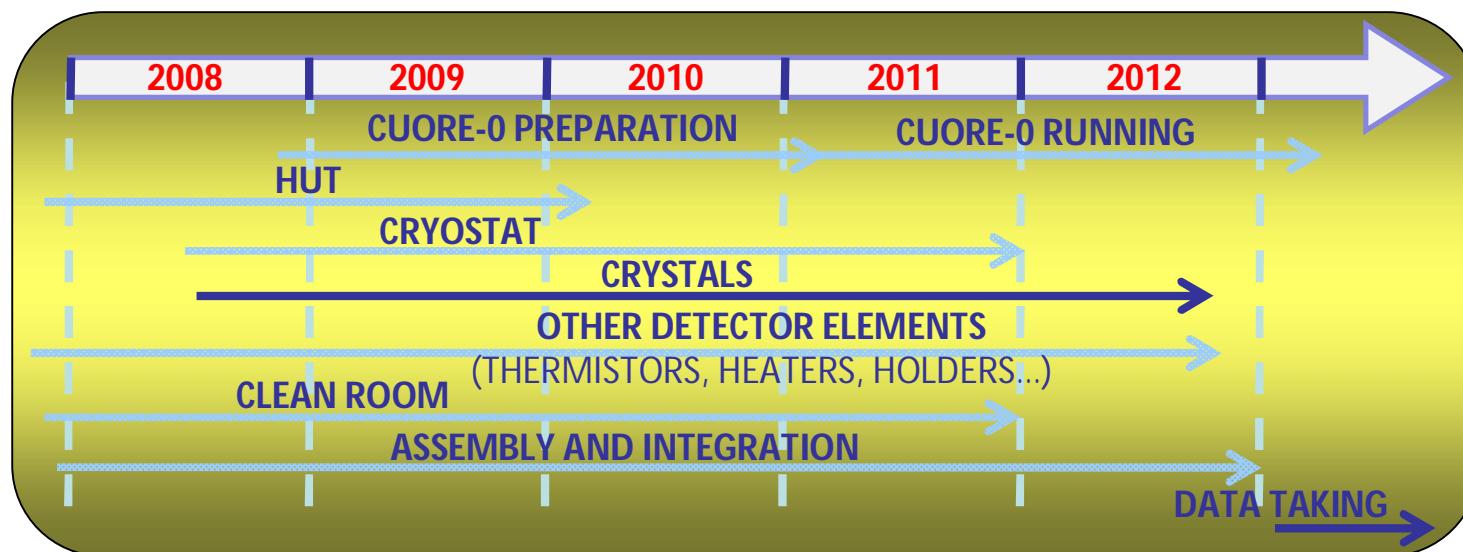
*-LabView GUIs generating .txt files
-crystal production data are further stored in a global CUORE database*



crystals production

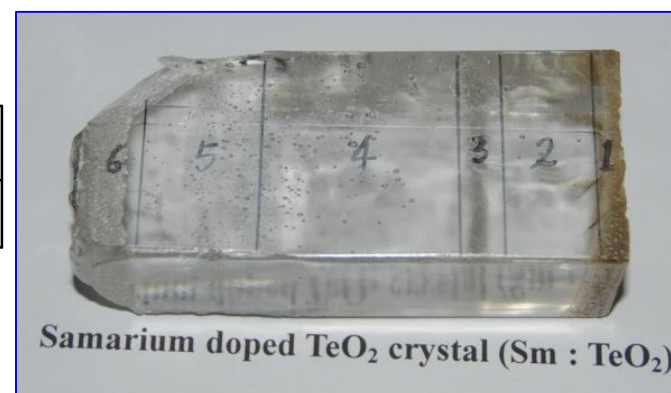


experiment timeline



- enriched TeO₂ ?
 - first tests, bad experience
- scintillating TeO₂ ?
 - *Nucl.Instr.Meth.in Phys.Res. A 554, Issues 1-3 , 1 December 2005, 195-200*
 - *SCINT2005 Proc. 8th Int. Conf., Alushta, Crimea, Ukraine, Sept. 19-23, 2005, pp. 106-108*
 - *phys. stat. sol. (a) 204, No. 5, 1567–1570 (2007) / DOI 10.1002/pssa.200622458*
- Sm doping?
 - calibration and possible discrimination of α events in the region of interest of ¹³⁰Te DBD0_v (2.5275 MeV)

isotope	abundance	half life	decay	energy	product
¹⁴⁷ Sm	14.99 %	1.06×10 ¹¹ y	α	2.310 MeV	¹⁴³ Nd



- TeO₂ crystal is among the best choices for DBD experiments
- growth and processing of TeO₂ crystals having a radio-purity compatible with foreseen CUORE total background of

0.01 counts/keV/kg/y

is a very challenging task

- TeO₂ crystals produced at SI CCAS (China) till present have bolometric and radio-purity characteristics within tolerance limits imposed by CUORE