



# Session on Enrichment/Purification/ Crystal Pulling

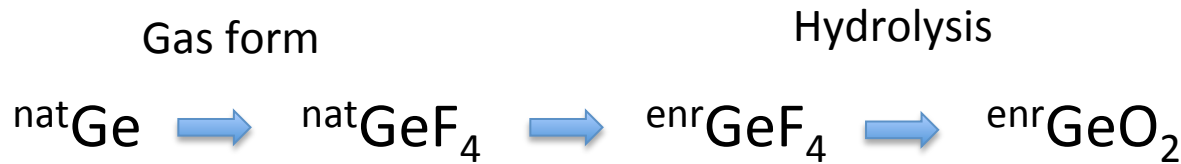
Introduction

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# Enrichment

## Enrichment



Centrifugal  
enrichment

Most expensive single step in  
production of Ge detectors is  
enrichment. Look into global  
possibilities.

## Plenty of Ge

### World Refinery Production and Reserves:

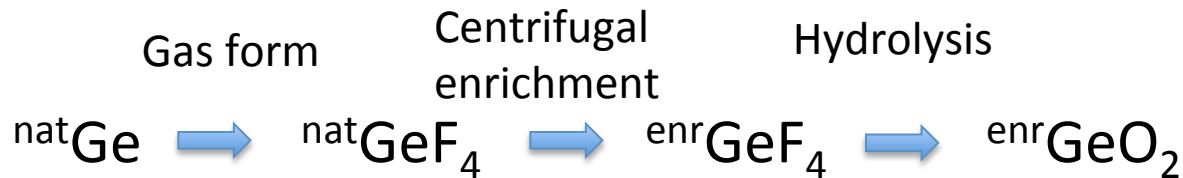
	Refinery production <sup>e</sup>		Reserves <sup>5</sup>
	<u>2014</u>	<u>2015</u>	
United States	W	W	Data on the recoverable content of zinc ores are not available.
China	120,000	120,000	
Russia	5,000	5,000	
Other countries <sup>6</sup>	40,000	40,000	
World total	<u>165,000</u>	<u>165,000</u>	

**U.S. Geological Survey, Mineral Commodity Summaries, January 2016**



# Enrichment

## Enrichment



	70	72	73	74	76
Nat Ge	20.54	27.4	7.76	36.54	7.76
Enriched Ge	0.015	0.075	0.165	12.5	87.25
$^{68}\text{Ge}$ Activation rate /kg/day	281	55.3	28.0	14.5	4.22
$^{60}\text{Co}$ Activation rate /kg/day	1.73	2.88	3.14	3.35	3.31

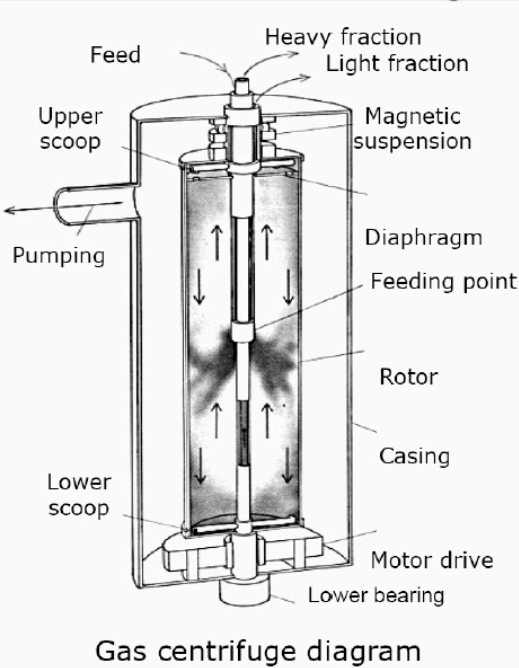
$^{68}\text{Ge}$  and  $^{60}\text{Co}$  are background sources for experiment. Enrichment used to reduce light isotopes of Ge to slow cosmogenic production of these isotopes.

$^{68}\text{Ge}$  accumulated after enrichment;  $^{60}\text{Co}$  after crystal pulling.

$T^{1/2}(^{68}\text{Ge}) = 270$  days;  $T^{1/2}(^{60}\text{Co}) = 5.27$  years

# Enrichment

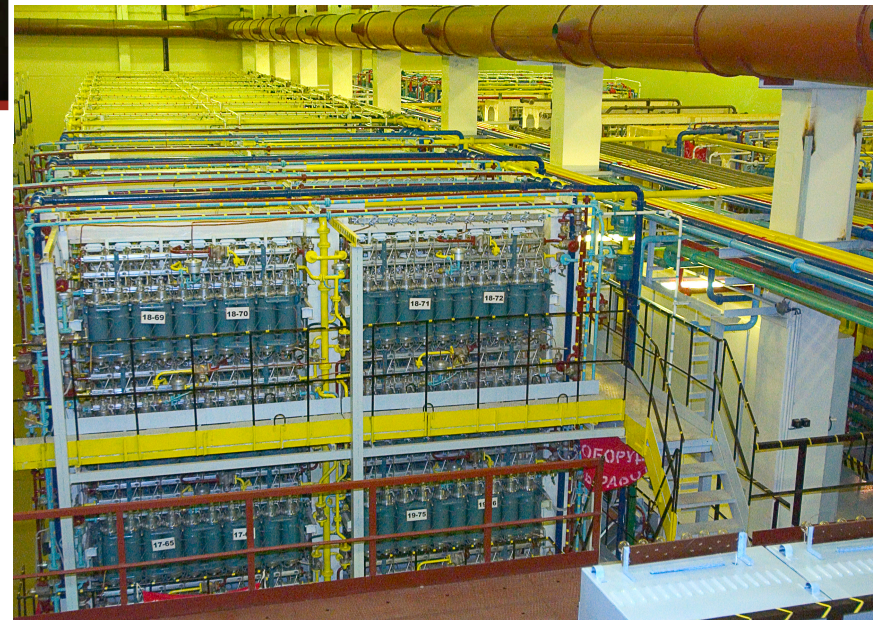
## Centrifuge separation



Gas centrifuge diagram

g. 6. Principle of gas-centrifuge isotope separation (G.Yu. Grigoriev, 2003).

ECP Plant  
Zhelenogorsk, Russia



Need cascade of centrifuges – months-long process. Throughput/year ?



# Transport

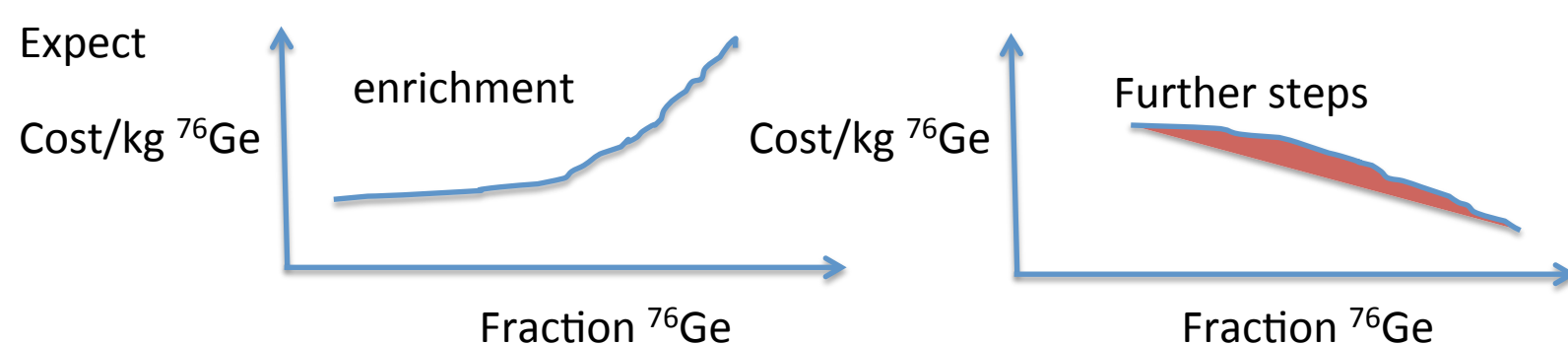




# Enrichment

In the following talks, we will hear about opportunities in Russia, USA and China. I will say a few words about Europe in the next slide.

Important consideration: what is the optimal level of enrichment, considering total cost, background, etc. ? Throughput at different sites ? Cleanliness of  $^{\text{enr}}\text{GeO}_2$  material ?



Generally expect background to increase with total mass of detectors; i.e., decrease with higher enrichment. Some active background suppression techniques improve (anticoincidence).

# Enrichment in Europe



Bernhard has been in discussions with URENCO, including paying them a visit.

Dear Mr. Schwingenheuer,

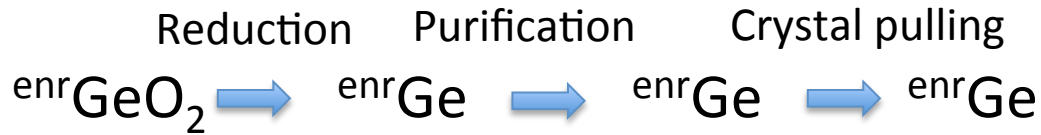
We are working on an expansion of our production capacity by the end of 2017. With this expansion, we will be able to reserve a part of our capacity for enrichment of  $76\text{GeF}_4$ . My expectations are that we should then be able to produce  $>60$  kg per year.

...

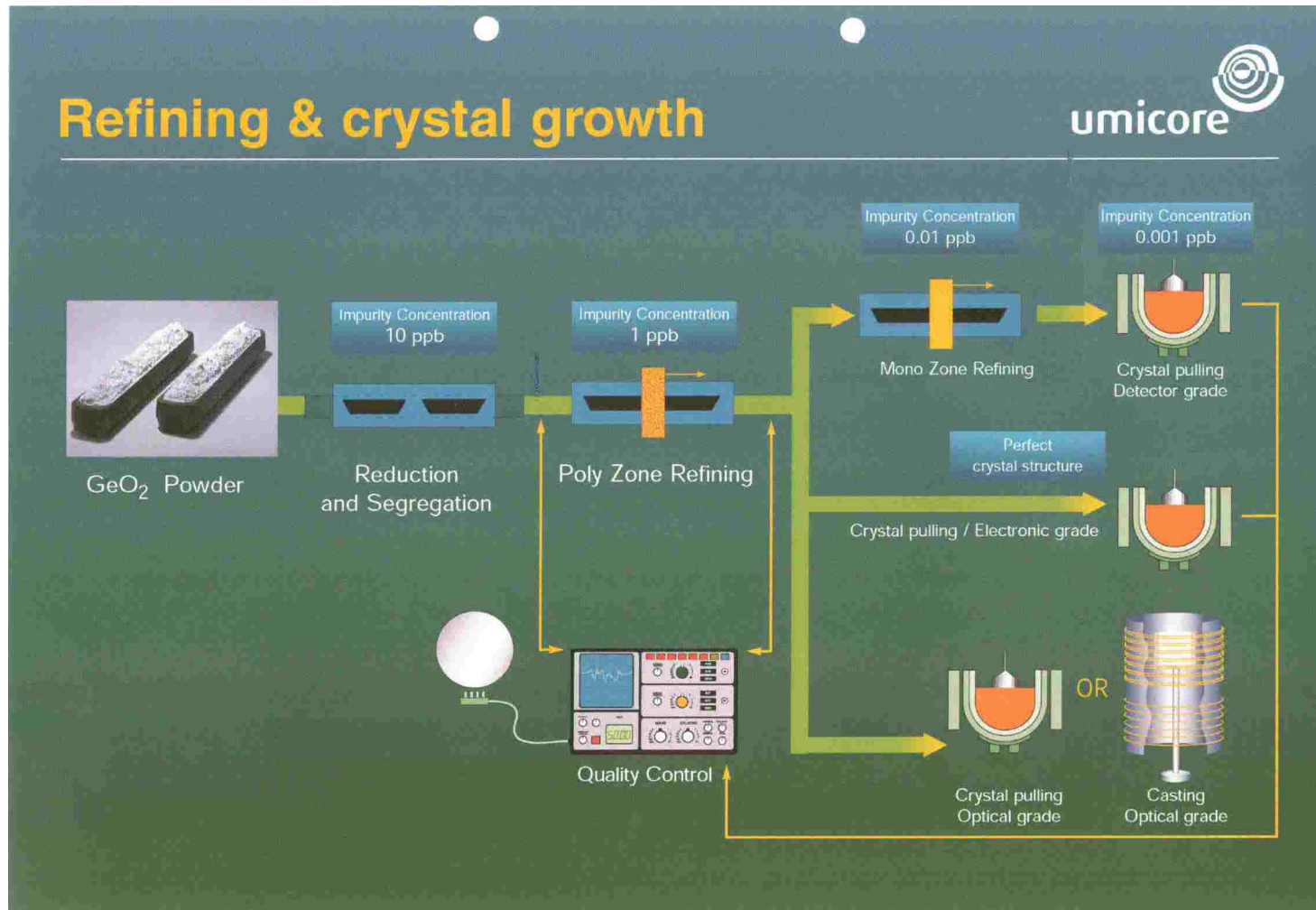
Possibly an interesting option !

# Purification & Crystal Pulling

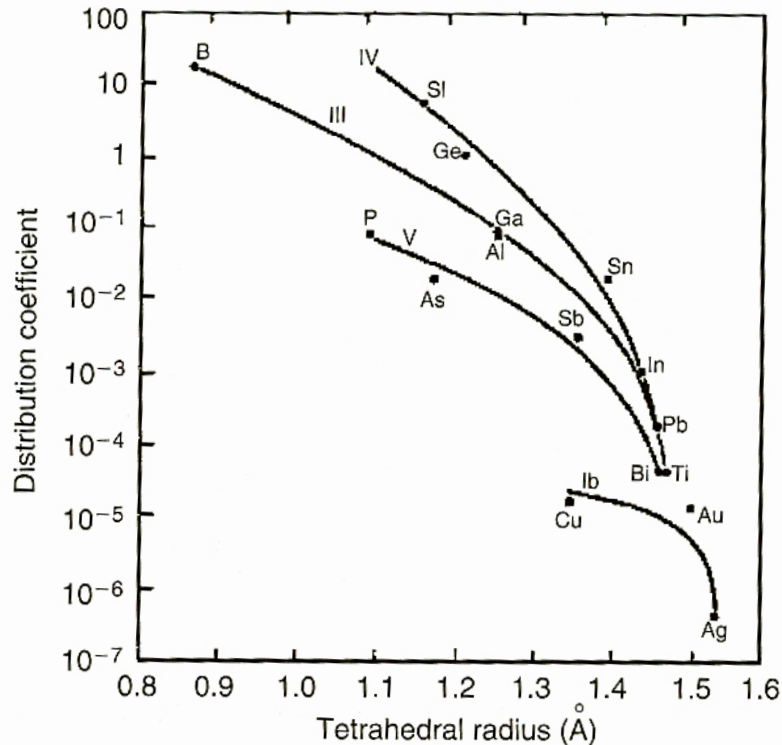
## Purification & xtal pulling



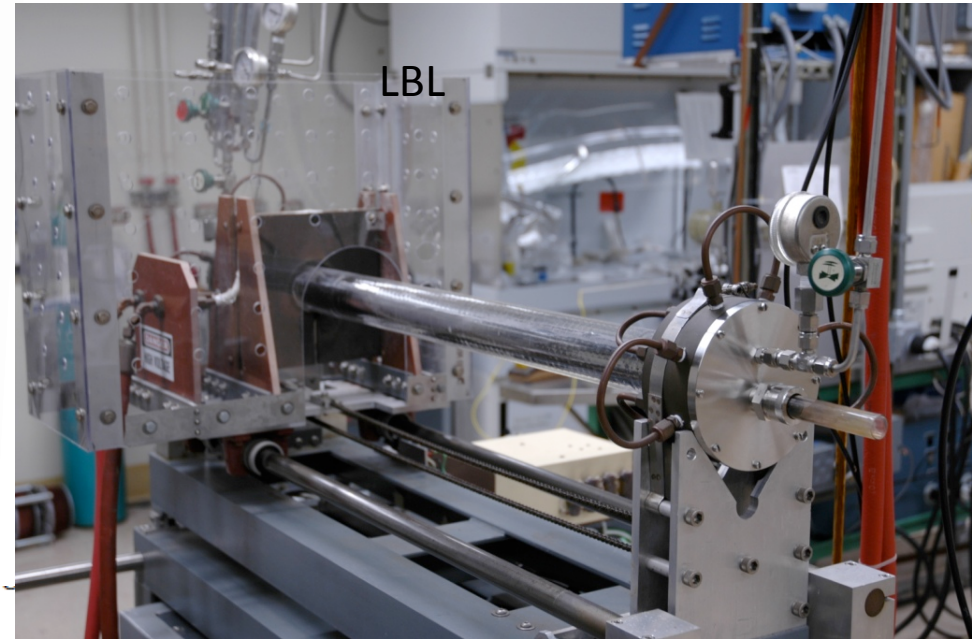
A lot of 'black magic' – particularly in crystal pulling to achieve the desired impurity concentrations.



# Purification



Zone refiner – pass melted zone across Ge bar

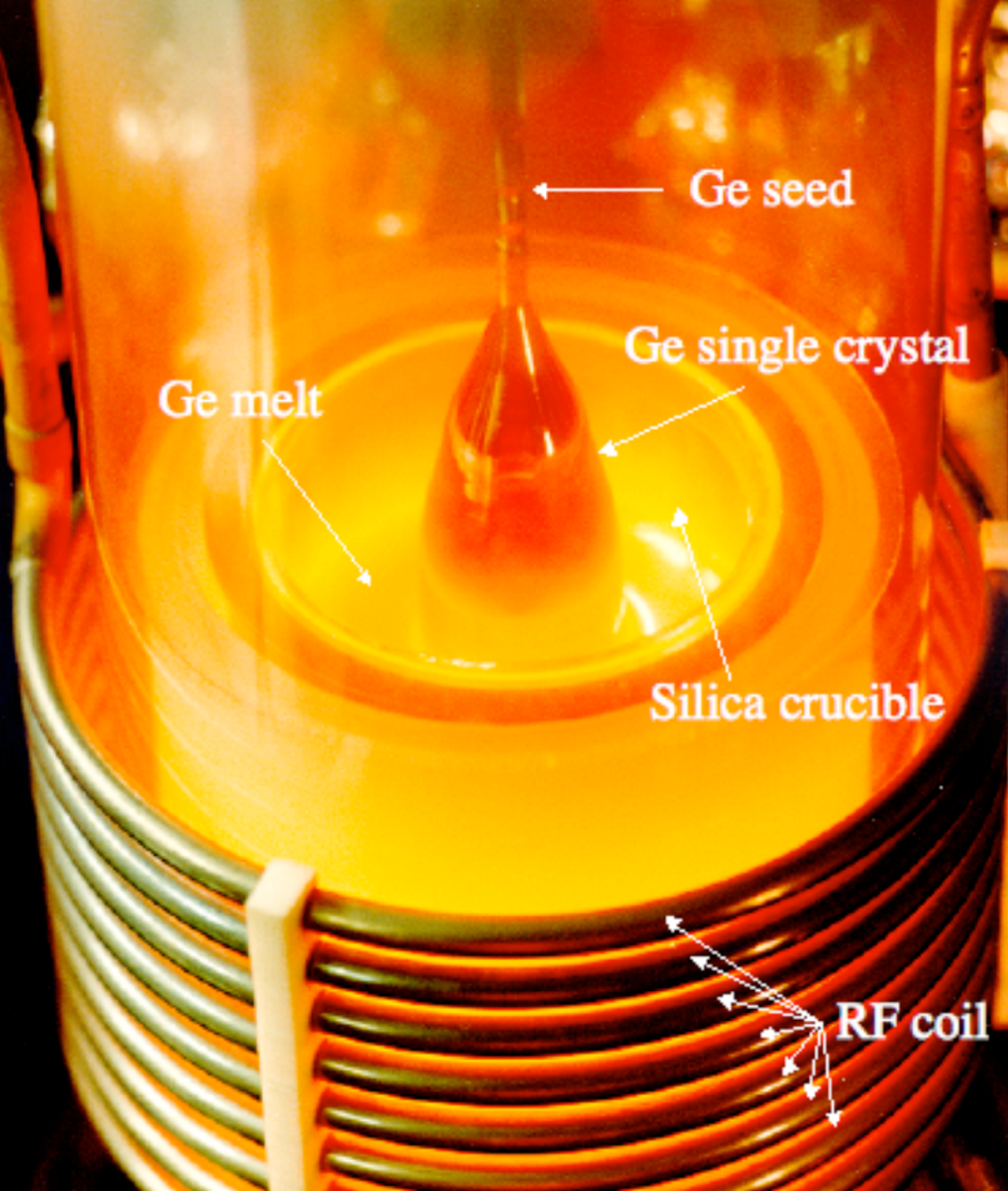


The segregation coefficient is defined as

$$(1) \quad k = \frac{C_S}{C_L}$$

where  $C_S$  and  $C_L$  are the impurity densities in the solid and liquid phase in atoms per cubic centimeter. The measured distribution coefficients are shown in Figure 1 (from Trumdore, *Bell Syst. Tech. J* 39 (1960) 169.) These are for equilibrium conditions between the melt and solid phase. The actual values achieved while pulling a crystal are considerably different, with e.g.  $k = 0.25, 1.0$  reported for P and Al in (E. Haller, W. Hansen, F. Goulding, *Advances in Physics* 30 (1981) 93.) This is due to finite pulling speed as well





An ultra-pure Germanium single crystal is being “pulled” from a melt contained in a silica crucible at  $936^{\circ}\text{C}$ . The atmosphere is pure Hydrogen. Heat is supplied by the water cooled radiofrequency (RF) coil surrounding the silica envelope. This bulk crystal growth technique carries the name of it’s inventor, “Jan Czochralski .”



# Purification & Crystal Pulling

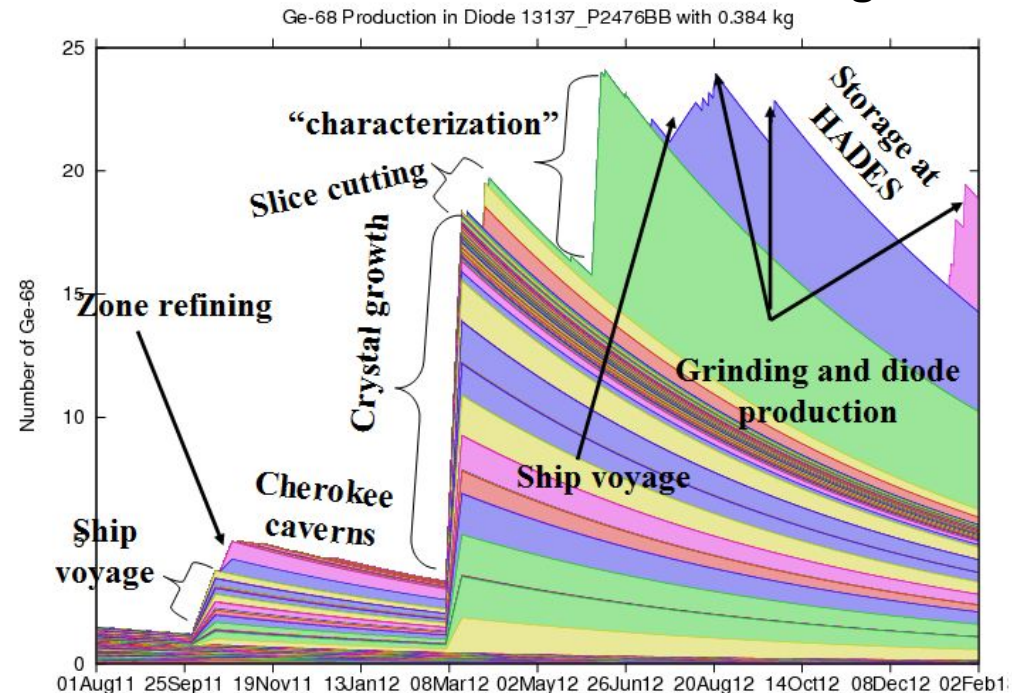
We will hear about opportunities in Europe, USA and China in the next talks.

Important consideration – transport logistics, background via activation, cost. About 50% of material ends up in useable crystals on first pass.

**Saturation concentration  
in  $^{enr}\text{Ge}$ :**

**$\sim 1600 \text{ }^{68}\text{Ge kg}^{-1}$ ,**

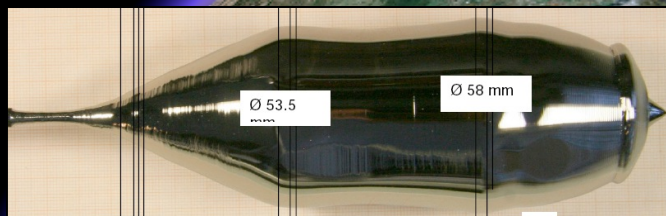
**$\sim 10.000 \text{ }^{60}\text{Co kg}^{-1}$**



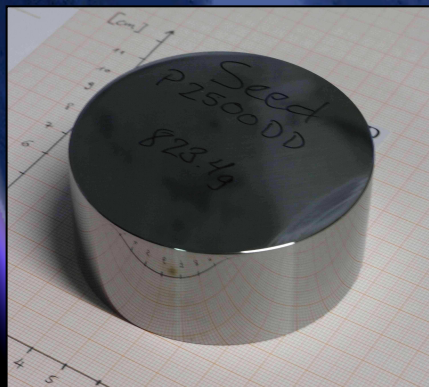




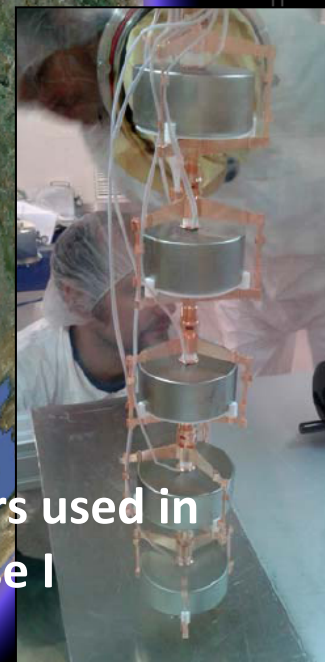
# Phase II – BEGe Production



Transports in  
shielded  
container,  
storage  
underground at  
HADES



→ 5 BEGe detectors used in  
GERDA Phase I



Data SIO, NOAA, U.S. Navy, NGA, GEBCO  
© 2012 Cnes/Spot Image  
Image © 2012 TerraMetrics

# Purification & Crystal Pulling

Some Considerations - enrichment

- Optimization: Cost vs degree of enrichment vs background expected
- Throughput at different facilities
- Logistics/transport

Some Considerations – Reduction/purification/crystal pulling/(det manufacture)

- Cost & throughput
- Logistics – do not want to ship material around the world for different steps
- Activation – is it a problem ? Should these steps be performed underground at location of experiment ?