

TG2 Status Report



1. ^{enr}Ge Transport
2. Isotopic composition
3. Purity measurements
4. Next steps

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Recall:

- 37.5 kg of ^{enr}Ge completed last September
- since then, several experiments to understand/improve yield of purification at Germaniy. Best result was about 75%.
- isotopic dilution was seen after purification step

The progress on improving the yield of the purification was not good, and the deadline on our Bank Certificate was approaching, so we decided to transport the material to Munich un-purified.

The ^{enr}Ge is in 5 batches: 1710-1714. A 3 gm sample was taken from each batch for quality tests

Transport of ^{enr}Ge

Times and actions:

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- 24.3.06** start transport ECP (Siberia)
- 3.4.06** at EU outside border
- 7.4.06** arrival at MPI
- 7.4.05** customs clearance OK, unpacking, weight determination, put back into PSP at MPI
- 10.4.06** MPI: PSP opening 13:30-14:00, loading storage boxes 14:00-14:40 departure to Geel (car, M.A+.K.K.) 14:55; arrival Geel 22:30
- 11.4.06** in HADES UGL: 9:30
- 11.4.06** 10:00: samples for MS taken from bags 8/11 (lot 1711) and bag 8/10 (lot 1714) (all from box-3); samples left with M.Hult
- 3.5.06** M.H. sub-divided samples, sent one sub-set via MPI to LNGS for ICP-MS



Check of sample weight

Celebrating with Vodka !



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**ПРОТОКОЛ ИСПЫТАНИЙ**  
 примесного состава №13388.06  
 Оксид германия (изотоп 76), проба №1710

Заказ №27/6-06 органа по Сертификации ООО "Ансертэко"

Отбор пробы осуществлен заказчиком.  
 Полный примесный и изотопный состав определяли методом искровой масс-спектрометрии на масс-спектрометре с двойной фокусировкой JMS-01-BM2, производства фирмы JEOL (Япония). Масс-спектры с высоким масс-спектральным разрешением регистрировали на фотопластины Pford-Q. Для количественной интерпретации масс-спектров применяли микроденситометр MDM6 производства фирмы Joaze Loebl (Великобритания), совмещенный с мини-ЭВМ NOVA 4 (США). При расчете содержаний примесей использовали математическое обеспечение, разработанное в MS & GC Lab. Случайная погрешность результатов анализа характеризуется значением относительного стандартного отклонения при определении изотопного состава 0,02, примесного состава 0,15-0,30. Содержания инертных газов и трансурановых элементов в данном партии находится ниже их пределов обнаружения - 0,001 ppm.

Результаты анализов представлены в массовых частях на миллион (1 ppm = 0.0001 %).

| Элемент | ppm масс | Элемент | ppm масс | Элемент | ppm масс |
|---------|----------|---------|----------|---------|----------|
| H       | ND       | Zn      | 2        | Pr      | < 0.02   |
| Li      | < 0.0006 | Ga      | < 0.04   | Nd      | < 0.05   |
| Be      | < 0.001  | Ge      | ОСНОВА   | Sm      | < 0.1    |
| B       | 0.2      | As      | < 0.04   | Eu      | < 0.04   |
| C       | 30       | Se      | < 0.02   | Gd      | < 0.2    |
| N       | 600      | Br      | < 0.06   | Tb      | < 0.03   |
| O       | ОСНОВА   | Rb      | < 0.03   | Dy      | < 0.1    |
| F       | < 0.1    | Sr      | < 0.02   | Ho      | < 0.03   |
| Na      | 0.6      | Y       | < 0.03   | Er      | < 0.04   |
| Mg      | 1        | Zr      | < 0.05   | Tm      | < 0.03   |
| Al      | 2        | Nb      | < 0.1    | Yb      | < 0.1    |
| Si      | 30       | Mo      | < 0.04   | Lu      | < 0.05   |
| P       | 8        | Ru      | < 0.08   | Hf      | < 0.1    |
| S       | 2        | Rh      | < 0.04   | Ta      | ND       |
| Cl      | ND       | Pd      | < 0.08   | W       | < 0.2    |
| K       | 0.07     | Ag      | < 0.05   | Re      | < 0.1    |
| Ca      | 2        | Cd      | < 0.2    | Os      | < 0.2    |
| Sc      | < 0.01   | In      | < 0.03   | Ir      | < 0.1    |
| Ti      | < 0.01   | Sn      | < 0.1    | Pt      | < 0.2    |
| V       | < 0.01   | Sb      | < 0.05   | Au      | < 0.1    |
| Cr      | 0.7      | Te      | < 0.1    | Hg      | < 0.3    |
| Mn      | 0.04     | I       | < 0.05   | Tl      | < 0.05   |
| Fe      | 5        | Cs      | < 0.05   | Pb      | < 0.1    |
| Co      | < 0.05   | Ba      | < 0.06   | Bi      | < 0.04   |
| Ni      | 0.4      | La      | < 0.09   | Th      | < 0.04   |
| Cu      | < 0.1    | Ce      | < 0.05   | U       | < 0.04   |



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3 grams were taken from each batch before shipment for spectroscopy measurements. Certificate of impurity measurement on each batch.

Quality 3.5N

# Isotopic Measurements

Differences much larger than claimed uncertainties: **TIMS**  
**Sector 54, Moscow, ECP, LNGS**

| A | Batch | 1710       | 1711                | 1712       | 1713       | 1714                |
|---|-------|------------|---------------------|------------|------------|---------------------|
|   | 70    | 0.01 0.05  | 0.03 0.05<br>0.001  | 0.02 0.05  | 0.01 0.05  | 0.02 0.05<br>0      |
|   | 72    | 0.05 0.08  | 0.06 0.08<br>0.03   | 0.10 0.08  | 0.05 0.08  | 0.09 0.08<br>0.03   |
|   | 73    | 0.14 0.07  | 0.16 0.07<br>0.11   | 0.16 0.07  | 0.16 0.07  | 0.17 0.07<br>0.13   |
|   | 74    | 11.93 12.3 | 12.12 12.2<br>10.35 | 12.40 12.3 | 12.50 12.2 | 12.78 12.3<br>10.94 |
|   | 76    | 87.87 87.5 | 87.63 87.6<br>89.51 | 87.32 87.5 | 87.28 87.6 | 86.94 87.5<br>88.90 |

# Isotopic Dilution

Table

| Mass number | Isotopic composition, % |      |      |      |      |      |       |
|-------------|-------------------------|------|------|------|------|------|-------|
|             | Ge1                     | Ge2  | Ge3  | Ge4  | Ge42 | GeO2 | natGe |
| 70          | 21,2+/-0,1              | 21,2 | 21,2 | 21,1 | 21,2 | 21,2 | 19,5  |
| 72          | 29,4+/-0,1              | 29,3 | 29,4 | 29,5 | 29,5 | 29,5 | 27,1  |
| 73          | 8,61+/-0,05             | 8,57 | 8,56 | 8,55 | 8,52 | 8,53 | 7,87  |
| 74          | 40,2+/-0,1              | 40,2 | 40,2 | 40,2 | 40,4 | 40,2 | 37,9  |
| 76          | 0,64+/-0,05             | 0,64 | 0,64 | 0,64 | 0,53 | 0,64 | 7,7   |

## Remarks:

Samples Ge1 – Ge4 are samples of reguluses (Ge ingots) produced by means of the same boat;  
Sample 42 – <sup>76</sup>freeGeO<sub>2</sub> from Svetlana (sampling made in Germaniy);  
Sample GeO<sub>2</sub> – <sup>76</sup>freeGeO<sub>2</sub> just after purification in FSUE Germaniy;  
NatGe – sample of natural germanium from FSUE Germaniy (as reference one).

Main conclusion: “Dilution” takes place during chemistry procedure. No isotopic “dilution” effect during reduction procedure.

**Note: different conclusion to previous**

Isotopic analysis of <sup>76</sup>freeGeO<sub>2</sub> and <sup>76</sup>freeGe samples after 3d experiment in FSUE Germaniy (Krasnoyarsk) with TIMS MS Sector-54

# TG2 Schedule

- Goal of the TG is to produce Phase II detectors for GERDA
- Achieved:
  - Procurement and transport of 37.5 kg of  $^{enr}\text{Ge}$  with isotopic content ca 87%
  - Set up of test facilities at MPI
  - Procurement and successful test of a prototype 18-fold segmented  $^{nat}\text{Ge}$  detector
- To be achieved:
  - operation of segmented detector in LAr
  - purification
  - crystal pulling
  - detector manufacture



# TG2 Schedule

- Plan:
  - We start by assuming Canberra-France will produce detectors (Ortec expressed no interest). Need a crystal source acceptable for Canberra.
  - Continue to try to get crystals from standard sources (Umicore, Tennelec)
  - In parallel, research other options:
    - discussions started with TU, Institut für Kristallzüchtung Berlin
  - If standard source:
    - time scale defined by crystal manufacturer (no start possible before Nov, + ca 6 months). Would need to settle purification first, but assume this is simpler.
  - If new source, will need a few iterations with  $^{\text{nat}}\text{Ge}$  first. 2+ years. Purification needs to be defined by crystal puller.