RPSCINT2013

“Production and deep purification of isotopically-enriched materials for $^{40}Ca^{100}MoO_4$ crystal growing

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ITEP&Fomos Materials
(Moscow, Russia)

Kiev, September 17 - 19, 2013

Items for discussion:

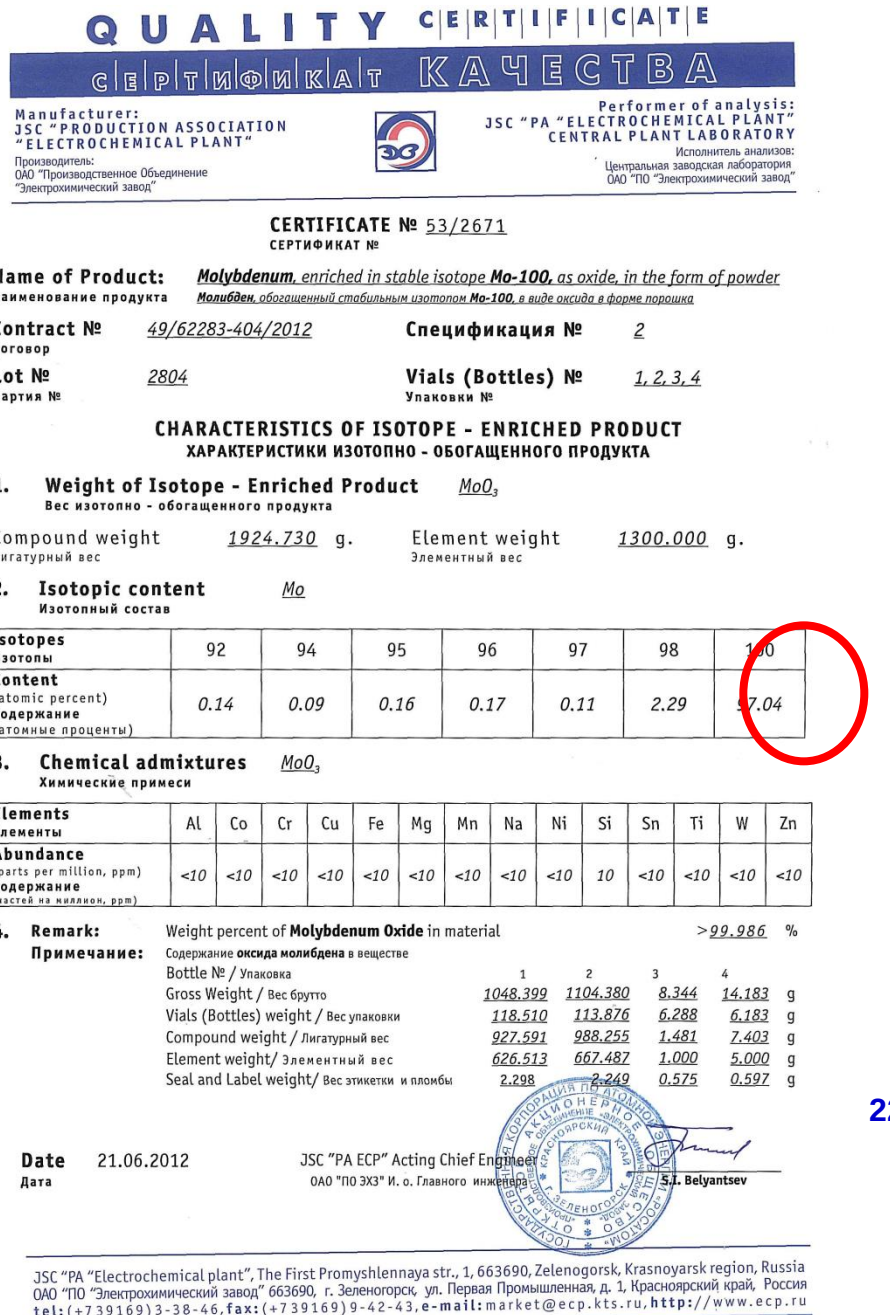
1. ECP (Zelenogorsk): Mo-100 production

2. EKP (Lesnoy): Ca-40 production and purification of Ca-40 compounds

3. NEOKHIM (Moscow): further purification of Mo-100 and Ca-40 and $^{40}Ca^{100}MoO_4$ raw material synthesis.

4. Decomposition of $^{40}Ca^{100}MoO_4$ waste

Mo-100



QUALITY CERTIFICATE

СЕРТИФИКАТ КАЧЕСТВА

Manufacturer:
JSC "PRODUCTION ASSOCIATION "ELECTROCHEMICAL PLANT"
Производитель:
ОАО "Производственное Объединение "Электрохимический завод"

Performer of analysis:
JSC "PA "ELECTROCHEMICAL PLANT" CENTRAL PLANT LABORATORY
Исполнитель анализов:
Центральная заводская лаборатория ОАО "ПО "Электрохимический завод"

CERTIFICATE № 53/2671
СЕРТИФИКАТ №

Name of Product: *Molybdenum, enriched in stable isotope Mo-100, as oxide, in the form of powder*
Наименование продукта *Молибден, обогащенный стабильным изотопом Mo-100, в виде оксида в форме порошка*

Contract № *49/62283-404/2012* **Спецификация №** *2*
Договор

Lot № *2804* **Vials (Bottles) №** *1, 2, 3, 4*
Партия № Упаковки №

CHARACTERISTICS OF ISOTOPE - ENRICHED PRODUCT
ХАРАКТЕРИСТИКИ ИЗОТОПНО - ОБОГАЩЕННОГО ПРОДУКТА

1. **Weight of Isotope - Enriched Product** *MoO_3*
Вес изотопно - обогащенного продукта

Compound weight *1924.730* g. Element weight *1300.000* g.
Лигатурный вес Элементный вес

2. **Isotopic content** *Mo*
Изотопный состав

Isotopes Изотопы	92	94	95	96	97	98	100
Content (atomic percent) Содержание (атомные проценты)	0.14	0.09	0.16	0.17	0.11	2.29	97.04

3. **Chemical admixtures** *MoO_3*
Химические примеси

Elements Элементы	Al	Co	Cr	Cu	Fe	Mg	Mn	Na	Ni	Si	Sn	Ti	W	Zn
Abundance (parts per million, ppm) Содержание (частей на миллион, ppm)	<10	<10	<10	<10	<10	<10	<10	<10	<10	10	<10	<10	<10	<10

4. **Remark:** Weight percent of **Molybdenum Oxide** in material *>99.986* %
Примечание: Содержание **оксида молибдена** в веществе

Bottle № / Упаковка	1	2	3	4	
Gross Weight / Вес брутто	1048.399	1104.380	8.344	14.183	g
Vials (Bottles) weight / Вес упаковки	118.510	113.876	6.288	6.183	g
Compound weight / Лигатурный вес	927.591	988.255	1.481	7.403	g
Element weight/ Элементный вес	626.513	667.487	1.000	5.000	g
Seal and Label weight/ Вес этикетки и пломбы	2.298	2.249	0.575	0.597	g

Date 21.06.2012 JSC "PA ECP" Acting Chief Engineer S.I. Belyantsev
Дата ОАО "ПО ЭХЗ" И. о. Главного инженера

JSC "PA "Electrochemical plant", The First Promyshlennaya str., 1, 663690, Zelenogorsk, Krasnoyarsk region, Russia
ОАО "ПО "Электрохимический завод" 663690, г. Зеленогорск, ул. Первая Промышленная, д. 1, Красноярский край, Россия
tel:(+739169)3-38-46, fax:(+739169)9-42-43, e-mail:market@ecp.kts.ru, http://www.ecp.ru

Total quantity of $^{100}MoO_3$ produced for R&D $^{40}Ca^{100}MoO_4$:

In 2008-2009: 2,5 kg

In 2012 -2013: 5,75 kg

IE = 97,04%

Impurities **(ICP MS measurements):**

U ≤ 0,1 ppb

Th ≤ 0,1 ppb

HPGe at Baksan:

^{226}Ra < 2,3 mBq/kg, ^{228}Ac < 3,8 mBq/kg

Chemistry is OK!

Production capacity of Mo-100 at the ECP

The working gas for Moly enrichment (MoF6) is extremely corrosive: once a machine is dedicated to Moly enrichment, there is no going back. You simply scrap the machine when the program is completed.

Because of worldwide shortage of Mo-99 for Tc99m generator production:

New proved technology: production of Mo-99 in the activation reaction:

98Mo(n,g)99Mo (reactor) and **100Mo(gamma,n)99Mo (e-linac)**

As result, current productivity is about 2,4 kg of Mo-100 per month ~ 28 kg per year

Ca-40 isotope production: ELEKTROCHIMPRIBOR (Lesnoy, Sverdlovky region)

Total quantity of $^{40}CaCO_3$ produced for R&D :

1.25 kg +3,2 kg (Ca-40)

Isotopic composition:

40 Ca – 99,96% - 99,988 % at.

46 Ca - < 0,001% at. % **(K depletion is 187 times)**

Impurities (the results ICP MS measurements):

U	≤ 0,1 ppb
Th	≤ 0,1 ppb
Sr	= 1 ppm
Ba	= 1 ppm
Ra-226	= 51 mBq/kg
Ac-228(Th-228)	= 1 mBq/kg

The industrial separator SU20
Lesnoy, Sverdlovky region

26 kg of Ca-40 ($^{40}CaCO_3$)
is available now

at EKP, Lesnoy
Ca-48 < 0,001%
< 0,005%

During 2013 ÷ 21014
plus ~ 3 kg
(based upon orders for Ca-48)

Production of ^{40}Ca depleted on ^{48}Ca at EKP

- Mechanical scraping of skims of receiving boxes with following treatment of the receiving boxes with different solvents + electrochemical stripping. Ca is transformed to solution by boiling procedure into acids w/o oxidants.
- Solution is evaporated and purified.
- Primary purification: recrystallization of **calcium oxalate** (precipitation as oxalate, filtration and washing with pure water):
- $(NH_4)_2C_2O_4 + CaCl_2 \rightarrow CaC_2O_4\downarrow + 2NH_4Cl$
- Then dissolution with HCl acid and filtration:
- $CaC_2O_4 + 2HCl \rightarrow H_2C_2O_4 + CaCl_2$
- **In total: 3 recrystallizations**.
- Precipitation as calcium carbonate (with ammonia carbonate):

$$(NH_4)_2CO_3 + CaCl_2 \rightarrow CaCO_3 + 2NH_4Cl$$

or

- $Ca + HCOOH \rightarrow Ca(HCOO)_2 \rightarrow$ recrystallization
- *Very last stages of purification are under hood w/o ventilation to prevent isotopic dilution (Ca is very common element).*

Progress in purification of Ca-40 at EKP (Lesnoy) and NEOKHIM (Moscow)

Isotope	Energy, (keV)	Activity, (Bq/kg)			
		EKP			NEOKHIM
		Sample №1 November 2009 standard technology	Sample №2 November 2010 purified as $CaCO_3$	Sample №3 October 2011 purified as $Ca(HCOO)_2$	Sample №3 December 2012 purified as $Ca(HCOO)_2$
^{40}K	1460,8	$(7,3+/- 3,1)\cdot10^{-2}$	$\leq 1,24\cdot10^{-2}$	$(3,6 \pm 2,7)\cdot10^{-3}$	$\leq 8,6\cdot10^{-3}$
^{208}Tl [Th-228]	2614,5	$(4,4+/- 3,6)\cdot10^{-3}$ [$(1,2+/- 1,0)\cdot10^{-2}$]	$(1,1 \pm 0,1)\cdot10^{-2}$ [$(3,0+/- 0,3)\cdot10^{-2}$]	$\leq 5,2\cdot10^{-4}$	$\leq 1,3\cdot10^{-3}$
^{214}Bi	609,3	$(2,6+/- 0,2)\cdot10^{-1}$ **260 mBq/kg**	$(1,72 \pm 0,05)\cdot10^{-1}$	$(5,1 \pm 0,2)\cdot10^{-2}$ **51 mBq/kg**	$(1,4 \pm 0,9)\cdot10^{-3}$ **1,4 mBq/kg**
^{228}Ac [Ra-228]	911,2	$(1,6+/- 0,2)\cdot10^{-1}$	$(5,9 \pm 0,5)\cdot10^{-2}$	$(1,0 \pm 0,8)\cdot10^{-3}$	$(1,3 \pm 1,1)\cdot10^{-3}$

Synthesis of $CaMoO_4$ raw material at NeoChem (Moscow)

Co-precipitation reaction:

Ca-compound (calcium formate):

$$(NH_4)_6Mo_7O_{24} + 7\ Ca(HCOO)_2 + 8\ NH_4OH \rightarrow$$
$$\rightarrow 7\ CaMoO_4 + 14\ NH_4(HCOO) + 4\ H_2O$$

- guaranteed stechiomery
- additional purification in the process
- NH_4OH and NO_3 are easy can removed by washing and heat treatment

Deep purification of isotopically-enriched components before $^{40}Ca^{100}MoO_4$ synthesis

- **Ca as formate**

Ca + HCOOH→ $Ca(HCOO)_2$ → recrystallization

  - Dissolving into в HNO_3
  - Crystallization into NH_4OH

- **Mo**
  - complexing agent (diethyl carbonate ditiocarbamate)
  - sorbent (activated charcoal/carbon)

Multistage purification of calcium formate before $^{40}Ca^{100}MoO_4$ synthesis

- Initial calcium formate is dissolved into HNO_3 and filtrated. Then formic acid is added, and very slowly - portion by portion - small portions of liquid ammonia are added (to neutralize nitric acid). Pulp of formate crystals is filtrated and carefully “squeezed” from “mother waters”.
- 2d and 3d processes (if necessary) are carried out
- Reagents of “o.c.ч.” grade, labware made of polymers.
- Final product before synthesis is in form of “fat/pregnant” solution.
- Ra226: 51 mBq/kg → 1,4 mBq/kg (additional purification of 3,6 times compare with EKP).

Purification of “ammonia paramolybdate” $(NH_4)_6Mo_7O_{24}$

- **Two-stages process**:

a) Dissolution of $(NH_4)_6Mo_7O_{24}$ into weak ammoniac solution and precipitation as molybdenum acid.

b) Dissolution of molybdenum acid into liquid ammonia, purification of the solution by sorption with complexing agent, filtration and evaporation with following crystallization of $(NH_4)_6Mo_7O_{24}$.

Before synthesis of raw material, $(NH_4)_6Mo_7O_{24}$ again is dissolved into weak ammoniac solution.

Details of $^{40}Ca^{100}MoO_4$ synthesis

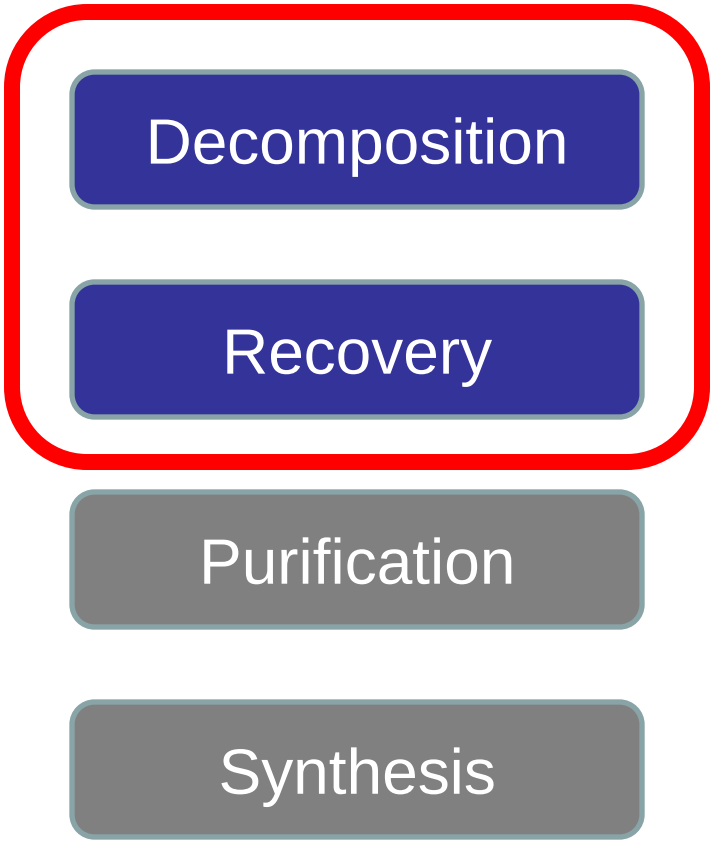
- Under heating (till 50 ÷ 60°C) to a solution of ammonia molybdate ones add – very slowly, portion by portion - a solution of calcium formate. Calcium molybdate is precipitated, and then the deposit is filtrated and washed up with pure water.
- $^{40}Ca^{100}MoO_4$ is placed into crucible made of quartz and heated under temperature 800°C during 4 hours.

Content of impurities into $^{40}Ca^{100}MoO_4$ raw material (measurements at EKP).

Элемент	Содержание, вес.%	Элемент	Содержание, вес.%	Элемент	Содержание, вес.%	Элемент	Содержание, вес.%
Li	<0.0001	Zn	<0.0002	Sn	<0.0001	Yb	<0.0001
Be	<0.0005	Ga	<0.0001	Sb	<0.0001	Lu	<0.0001
B	<0.001	Ge	<0.0001	Te	<0.0002	Hf	<0.0001
Na	0.003	As	<0.0001	I	<0.0005	Ta	<0.0001
Mg	<0.0003	Se	<0.002	Cs	<0.0001	W	<0.0001
Al	<0.0003	Br	<0.005	Ba	<0.0001	Re	<0.0001
Si	<0.005	Rb	<0.0001	La	<0.0001	Os	<0.0001
P	<0.005	Sr	<0.0001	Ce	<0.0001	Ir	<0.0001
K	<0.005	Y	<0.0001	Pr	<0.0001	Pt	<0.0001
Sc	<0.0002	Zr	<0.0001	Nd	<0.0001	Au	<0.0001
Ti	<0.0004	Nb	<0.0001	Sm	<0.0001	Hg	<0.0001
V	<0.0004	Mo	<0.0001	Eu	<0.0001	Tl	<0.0001
Cr	<0.001	Ru	<0.0001	Gd	<0.0001	Pb	0.0001
Mn	0.0001	Rh	<0.0001	Tb	<0.0001	Bi	<0.0001
Fe	<0.005	Pd	<0.0001	Dy	<0.0001	Th	<0.0001
Co	<0.0001	Ag	<0.0001	Ho	<0.0001	U	<0.0001
Ni	<0.0001	Cd	<0.001	Er	<0.0001		
Cu	<0.0001	In	<0.0001	Tm	<0.0001		

General scheme of regeneration process:

Pavel Moseev, Fomos Materials, AMORE meeting, Moscow 9 - 10 of July 2012



Decomposition with inorganic acids

- High solubility of molybdenic acid in concentrated hydrochloric as a result of formation of a complex :

$$CaMoO_4 + 6HCl = CaCl_2 + H_2MoCl_4O_2 + 2H_2O$$

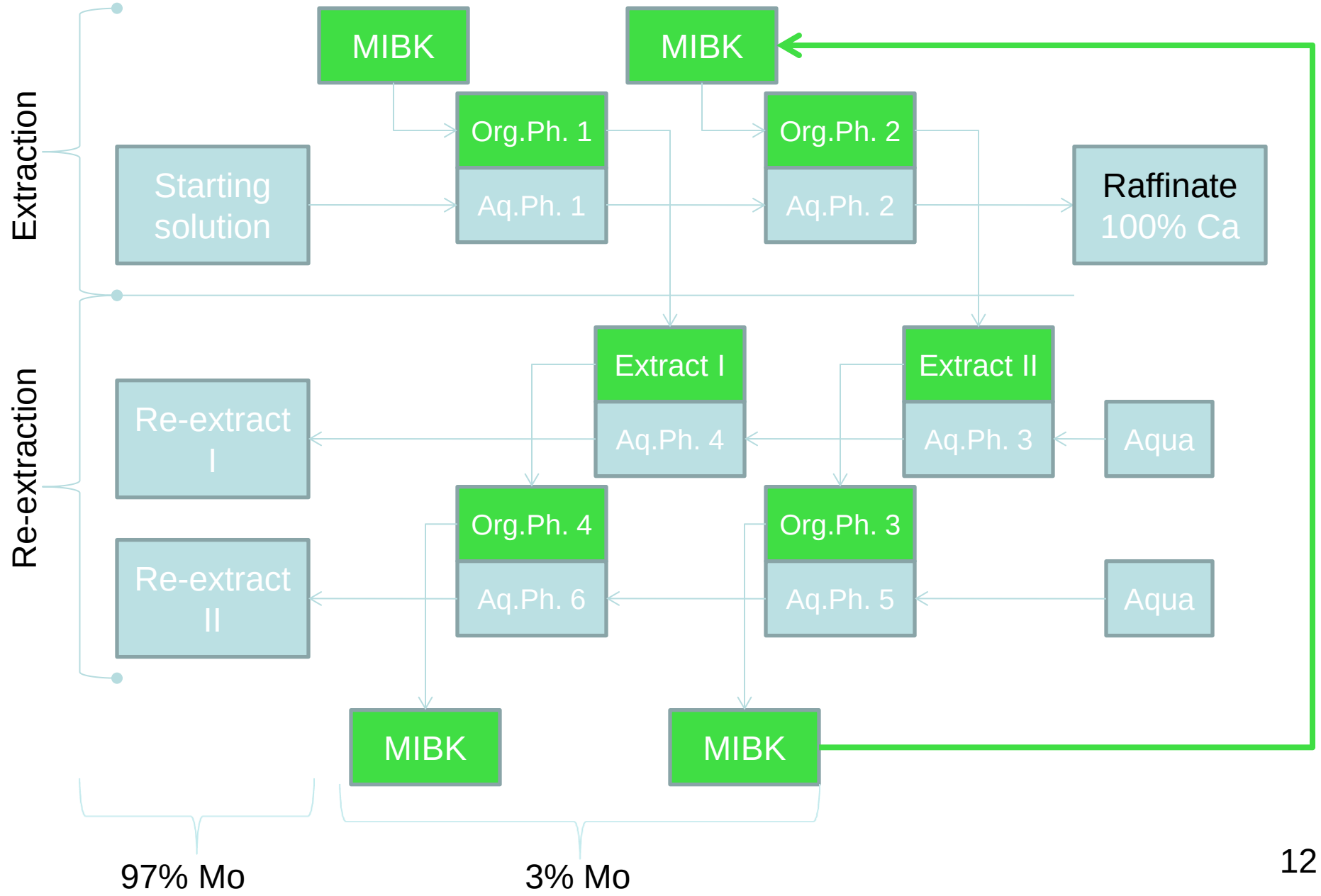
- The complete dissolution in 36% HCl is reached at a molar ratio 1:8 when boiling on a water bath within 30 minutes.

Molybdenum extraction by organic extractants.

Methylisobutyl ketone(MIBK): D=94 (C_{HCl} = 6M)

- Rather high distribution coefficient D
- Phase separation within 1 minute when re-extraction by water

Double extraction and double re-extraction scheme



Results

- Double extraction:
  - Yield of Ca – 100% (Raffinate)
  - Yield of Mo – 99,99% (Org.Ph.1 + 2)
- Double re-extraction by water allow to recover 97% of Mo (Re-extract I + II)
- 3% of Mo is in Org.Ph. used for next portion of starting solution

Recovery of Mo-100 and Ca-40 isotopes after decomposition of $^{40}Ca^{100}MoO_4$ waste

Recovery of Ca component from the solution

- Neutralization by liquid ammonia
- Add $(NH_4)_2CO_3$ to precipitate $CaCO_3$
- Filtration $CaCO_3$
- Retain/keep “mother” solution with Mo for further recovery procedure

- **Result:**
  - Ca is into highly/readily soluble $CaCO_3$
  - Mo is in form of H_2MoO_4 and into solution

Thank you!

Back up slides

Activity of radioactive isotopes (Bq / kg) (95% C.L.)
Baksan Neutrino Laboratory

Sample, material	Isotopes			
	^{40}K	$^{228}Ac=(^{232}Th)$	$^{208}Tl\ [(^{232}Th)]$	$^{214}Bi=(^{238}U)$
	Activity of radioactive isotopes			
Charge (raw material) $40Ca100MoO_4$	$\leq 9{,}4\cdot10^{-3}$	$(1{,}9\pm1{,}3)\cdot10^{-3}$	$\leq 1{,}1\cdot10^{-3}$	$\leq 1{,}6\cdot10^{-3}$
Calcium formate $40Ca(HCOO)_2$ (unpurified)	$\leq 3{,}4\cdot10^{-2}$	$\leq 9{,}1\cdot10^{-3}$	$\leq 8{,}3\cdot10^{-3}$	$(5{,}9\pm3{,}8)\cdot10^{-3}$
Calcium formate $40Ca(HCOO)_2$ (purified)	$\leq 8{,}6\cdot10^{-3}$	$(1{,}3\pm1{,}1)\cdot10^{-3}$	$\leq 1{,}3\cdot10^{-3}$	$(1{,}4\pm0{,}9)\cdot10^{-3}$

Separation of Mo and Ca components

- Direct reaction with HNO3 nitric acid:

$CaMoO4 + 2\ HNO_3 \rightarrow \mathbf{Ca(NO_3)_2} + H_2MoO_4$

- Pour solubility of H_2MoO_4 into water. Need to add peroxide:

$CaMoO_4 + 2\ HNO_3 + H_2O_2 \rightarrow Ca(NO_3)_2 + H_2MoO_5 + H_2O$

Complete dessolving

For separation of components (Mo and Ca) – boiling procedure

$H_2MoO_5\ (t^o) \rightarrow \mathbf{H_2MoO_4}\downarrow + ½\ O_2\uparrow$

Content of radioisotopes in single crystals

(HPGe measurements at Baksan Neutrino Observatory)

Sample, material	^{40}K	^{228}Ac=(^{232}Th)	^{208}Tl [(^{232}Th)]	^{214}Bi=(^{238}U)
	Activity of radioactive isotopes			
Monocrystal $CaMoO_4$ from B 19ББ	(2,0±0,8)×10-2	≤5,6×10-3	≤1,4×10-3 [≤3,9×10-3]	≤2,2×10-3
Monocrystal $CaMoO_4$ 29СБ	≤8,2×10-3	≤3,1×10-3	≤6,6×10-4 [≤1,8×10-3]	≤3,2×10-3
Monocrystal $CaMoO_4$ 42СБ	(1,2±0,6)×10-2	≤3,28×10-3	≤7,80×10^{-4} [≤2,15×10-3]	(1,6±0,2)×10-2
Monocrystal $CaMoO_4$ 42СБ	≤1,2· 10^{-2}	≤2,4· 10^{-3}	(9,4 ± 5,4)* 10^{-4}	(9,5 ± 2,2)* 10^{-3}
Monocrystal $CaMoO_4$ 48ББ	≤1,4×10^{-2}	(2,3±1,9)×10^{-4}	(9,5±6,4)×10^{-4}	(1,0 ± 0,3)× 10^{-2}
Monocrystal $CaMoO_4$ 51ББ	≤1,2×10^{-2}	≤3,1×10^{-3}	(1,0±0,5)×10^{-3}	(1,0±0,3)×10^{-2}
Monocrystal $CaMoO_4$ 59ББ cut from one side	≤1,3×10^{-2}	≤3,4×10^{-3}	(5,0±4,9)×10^{-4}	≤5,3×10^{-3}

Sample, material	Form	Mass, g	Measurement time, h
Monocrystal $CaMoO_4$ from B 19ББ	Elliptical the cylinder with a convex top d_1=50mm, d_2=44mm, h » 40mm	384.5	498
Monocrystal $CaMoO_4$ 29СБ	Elliptical the cylinder with a convex top d_1=49mm, d_2=42mm, h » 71mm	553.5	795
Monocrystal $CaMoO_4$ 42СБ	Elliptical the cylinder with a convex top d_1=48mm, d_2=45mm, h » 95mm	494.87	893
Monocrystal $CaMoO_4$ 42СБ	Boule d=45,2mm, h=7mm	494.87	515
Monocrystal $CaMoO_4$ 48ББ	Boule (blue color) d_1=52/47mm,	657.83	323
Monocrystal $CaMoO_4$ 51ББ	Boule (white color) d_1=52/47mm, d_2=52/46mm,	661.28	482
Monocrystal $CaMoO_4$ 59ББ cut from one side	Boule (white color) d_1=52/47mm, d_2=52mm, h » 100/60mm	473.9	527

- ***Monocrystal $CaMoO_4$ from B 19ББ*** Без всяких добавок
- ***Monocrystal $CaMoO_4$ 29СБ*** добавлен оксид молибдена при выращивании перекриталлизата, использовали тигельные остатки, много процессов
- ***Monocrystal $CaMoO_4$ 42СБ*** добавлен МоО3 при выращивании перекриталлизата, использовали тигельные остатки, очень много аварийных процессов
- ***Monocrystal $CaMoO_4$ 48ББ*** оксид молибдена добавлен в шихту при выращивании первичного кристалла
- ***Monocrystal $CaMoO_4$ 51ББ*** Оксид ниобия добавлен в перекристаллизат при выращивании «финального» кристалла
- ***Monocrystal $CaMoO_4$ 59ББ*** *оксид ниобия добавлен в шихту при выращивании обоих первичных кристаллов*

Conclusions

- No doping!
- Transparence: annealing procedure&$^{100}MoO_3$ addition (stechiometry)
- FOMOS needs *Specifications approved by AMORE* for 40Ca100MoO4 crystals (LY (RT), LY (mK temperature), Transparency, Quality of phonon signal at mK, etc?)

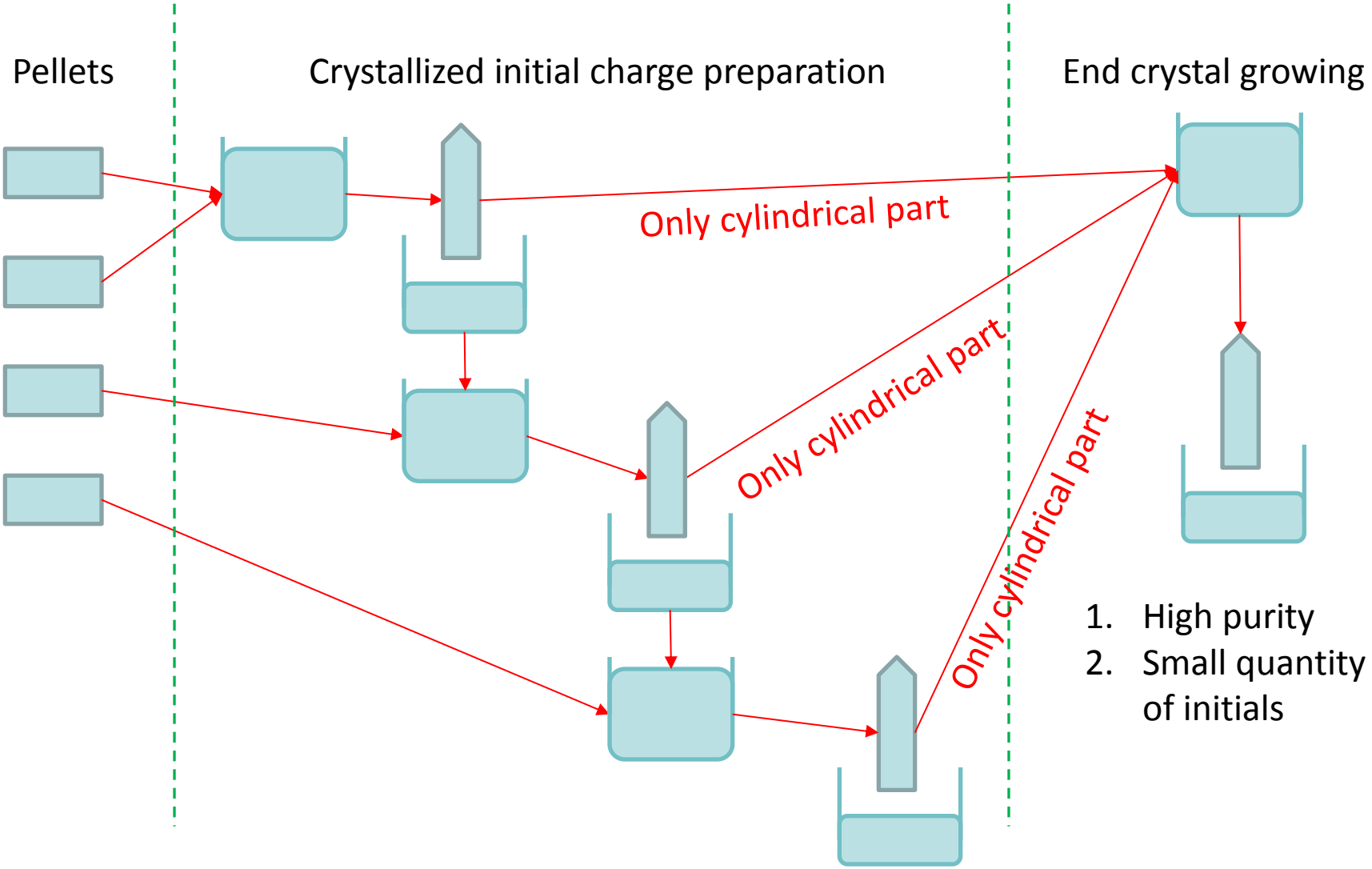
Activity of radioactive isotopes in the samples (Bq / kg) (95% C.L.)

Sample, material	Isotopes			
	^{40}K	$^{228}Ac=(^{232}Th)$	$^{208}Tl\ [(^{232}Th)]$	$^{214}Bi=(^{238}U)$
	Activity of radioactive isotopes			
Niobium pentoxide Nb_2O_5	$\leq 3{,}6\cdot10^{-2}$	$\leq 6{,}8\cdot10^{-3}$	$\leq 5{,}4\cdot10^{-3}$	$\leq 5{,}8\cdot10^{-3}$
Single crystal $CaMoO_4$	$\leq 1{,}4\cdot10^{-2}$	$(2{,}3\pm1{,}9)\cdot10^{-4}$	$(9{,}5\pm6{,}4)\cdot10^{-4}$	$(1{,}0\pm0{,}3)\cdot10^{-2}$
Singlecrystal $CaMoO_4$	$\leq 1{,}2\cdot10^{-2}$	$\leq 3{,}1\cdot10^{-3}$	$(1{,}0\pm0{,}5)\cdot10^{-3}$	$(1{,}0\pm0{,}3)\cdot10^{-2}$
Calcium formate $40Ca(HCOO)_2$ (purified)	$\leq 8{,}6\cdot10^{-3}$	$(1{,}3\pm1{,}1)\cdot10^{-3}$	$\leq 1{,}3\cdot10^{-3}$	$(1{,}4\pm0{,}9)\cdot10^{-3}$
Charge (raw material) $40Ca100MoO_4$	$\leq 9{,}4\cdot10^{-3}$	$(1{,}9\pm1{,}3)\cdot10^{-3}$	$\leq 1{,}1\cdot10^{-3}$	$\leq 1{,}6\cdot10^{-3}$
Single crystal $CaMoO_4$ BB-type	$\leq 1{,}3\cdot10^{-2}$	$\leq 3{,}4\cdot10^{-3}$	$(5{,}0\pm4{,}9)\cdot10^{-4}$	$\leq 5{,}3\cdot10^{-3}$
Calcium formate $40Ca(HCOO)_2$ (unpurified)	$\leq 3{,}4\cdot10^{-2}$	$\leq 9{,}1\cdot10^{-3}$	$\leq 8{,}3\cdot10^{-3}$	$(5{,}9\pm3{,}8)\cdot10^{-3}$

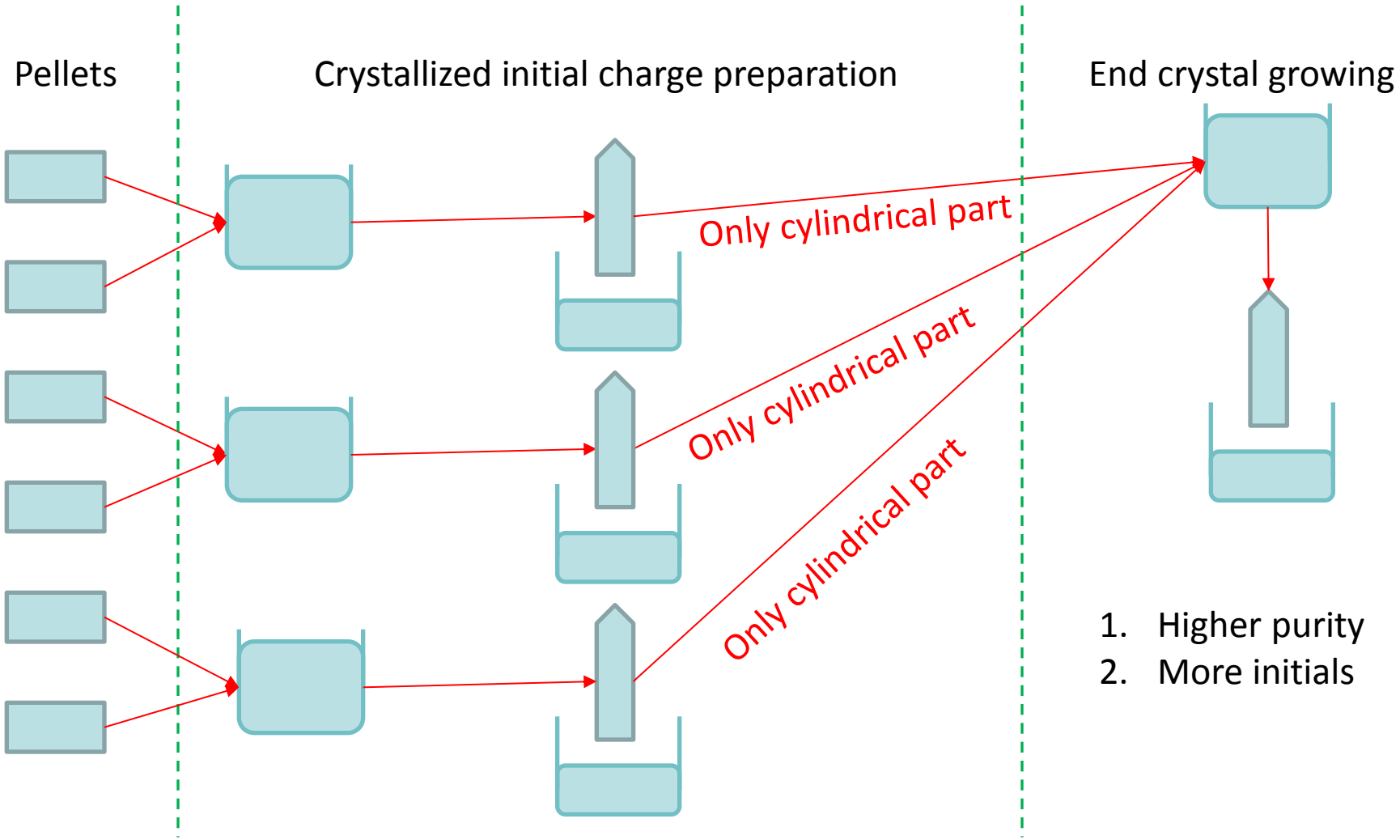
Fomos-Materials Co. crystal growth potential

- Fomos-Materials Co. has 15 crystal growth stations (6 established and 9 in stock)
- One crystal growth station can produce at least 18 end-crystals per year up to 600 g and 48 mm in diameter each.
- Fomos-Materials has 3 high temperature (up to 1600 dag. Cent) furnaces Carbolite 16/35 type for crystals heat treatment
- Fomos-Materials Co. has all technological equipment for crystal mechanical treatment (cutting, lapping, polishing)

Crystal growth process scheme
One-by-one



Crystal growth process scheme
Parallel



1. Higher purity
2. More initials

Characterization of the $^{40}Ca^{100}MoO_4$ samples

- Baksan Neutrino Observatory, Ra-226 at level:

  a) HPGe facilities 10 mBq/kg (powder-like samples) and 1,0 mBq/kg (singe crystal)

  b) New Low Background Facility (*active mode*): 0,1 mBq/kg

- EKP (Lesnoy, Sverdlovsk region), U-238&Th-232:

  ICP MS + U&Th pre-concentration: 0,1 – 0,01 ppb

- IMHPM RAS, KNU (Korea), Oxford University(UK): The LY at the range of 300 K – 9 K.
- Institute of Crystallography RAS:

  light transmittance at 520 nm

Образец, Материал, масса и время измерения	Изотоп			
	^{40}K	^{228}Ac (^{232}Th)	^{208}Tl (^{232}Th)*	^{214}Bi (^{226}Ra)
	Удельная активность, Бк/кг			
Мо оксид, $^{100}MoO_3$	$(5,3\pm0,8)\cdot10^{-2}$	$\leq3,8\cdot10^{-3}$	$\leq1,0\cdot10^{-3}$ [$\leq2,8\cdot10^{-3}$]	$\leq2,3\cdot10^{-3}$
Формиат кальция Очистка НЕОХИМ $^{40}Ca(HCOO)_2$	$\leq 8,6\cdot10^{-3}$	$(1,3 \pm 1,1)\cdot10^{-3}$	$\leq 1,3\cdot10^{-3}$	$(1,4 \pm 0,9)\cdot10^{-3}$
Шихта, $^{40}Ca^{100}MoO_4$	$\leq9,4\ 10^{-3}$	$(1,9 \pm 1,3)\ 10^{-3}$	$\leq 1,1^*\ 10^{-3}$	$\leq 1,6^*\ 10^{-3}$

2) Progress in purification of Ca-40 at EKP in 2009 - 2011

Элемент	$^{40}CaCO_3$ ВП-1-04 (11.04.2008 г.)			$^{40}CaCO_3$ ВП-2-10 (23.06.2010)	$^{40}Ca(HCOO)_2$ ВП-10-04-Э (25.01.2011)	Formate 3D and 4D $^{nat}Ca(HCOO)_2$
	DL, ppm	**IMHPM**	**EKP**	**EKP**	**EKP**	**EKP**
Sr	0,06	24÷27	23	7	**3**	**< 1**
Ba	0,05	24÷28	23	**7**	**1**	**< 1**
^{238}U	0,0002	< 0,0002	< 1	**< 1**	**< 1**	**< 1**
^{232}Th	0,0008	< 0,0008	< 1	**< 1**	**< 1**	**< 1**
	Baksan Neutrino Observatory, Bq/kg					
^{214}Bi (^{226}Ra)	$(2,6+/- 0,2)\cdot10^{-1}$			$(1,72 \pm 0,05)\cdot10^{-1}$	$(5,1 \pm 0,2)\cdot10^{-2}$	$\leq 1,7\cdot10^{-3}$
^{228}Ac (^{226}Ra)	$(1,6+/- 0,2)\cdot10^{-1}$			$(5,9 \pm 0,5)\cdot10^{-2}$	$(1,0 \pm 0,8)\cdot10^{-3}$	$\leq 3,0\cdot10^{-3}$
^{208}Tl [Th-228]	$(4,4+/- 3,6)\cdot10^{-3}$ [$(1,2+/- 1,0)\cdot10^{-2}$]			$(1,1 \pm 0,1)\cdot10^{-2}$ [$(3,0+/- 0,3)\cdot10^{-2}$]	$\leq 5,2\cdot10^{-4}$	$\leq 8,9\cdot10^{-4}$ $\leq 2,5\cdot10^{-3}$]

General scheme of the selection

- **$^{40}CaCO_3$, $^{100}MoO_3$ and $^{40}Ca^{100}MoO_4$ raw material**:

  U ≤ 0,1 ppb (≤ 1,2 mBq/kg)

  Th ≤ 0,1 ppb (≤0,4 mBq/kg)

  ^{226}Ra ≤ 10 mB/kg

  ⇓

- **$^{40}Ca^{100}MoO_4$ single crystal**:

  Absorption coefficient (520 nm): ≤ 0,011см$^{-1}$

  ⇓

- **$^{40}Ca^{100}MoO_4$ scintillation element (**Dimensions OK**)**:

  LY: ≥ 5 000 photon/MeV (300K)

  ≥ 10 000 photon/MeV(9 K)

  Specific activity of Ra-226 ≤ 1,0 ***(0,1)*** mBq/kg

  U < 0,01 ppb (≤ 0,12 mBq/kg)

  Th < 0,01 ppb (≤ 0,04 mBq/kg)

Requirements to $^{40}Ca^{100}MoO_4$ scintillation element

- **Cylindrical shape with elliptic(al) cross-section:**
- minor diameter: from 42 till 47 mm
- large diameter: from 47 till 52 mm,
- Height: from 40 till 50 mm

- **Light output** (small sample of 1 x 1 x 1 cm&Cs-137):
  ≥ 5 000 photon/MeV under RT (300K)
  ≥ 10 000 photon/MeV under 9 K

- **Specific activity of Ra-226** ≤ 1,0 mBq/kg
- **Uranium U** < 0,01 ppb
- **Thorium Th** < 0,01 ppb

- 1 ppb of U-238 = ~ 12 mBq/kg
- 1 ppb of Th-232 = ~ 4 mBq/kg
- ~ 1 mBq/kg = 0,081ppb of U-238 ~ 0,1 ppb
- ~ 1 mBq/kg = 0,247 ppb of Th-232 ~ 0,25 ppb

Further operation

- Precipitation $CaCO_3$ from the raffinate by adding $(NH_4)_2CO_3$. Yield ~100%.
- Mo is in form of H_2MoO_4 in Re-extract I + II ready to use for purification stage

Conclusion: This scheme provides the best yield and reasonable ease to perform