

**Conversion of some neutron-excess  
isotope chemical forms available at  
the market into compounds suitable  
for effective feeding of a cyclotron  
ECR ion source for production  
highly intense ion beams**

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**Production of highly intense ion beams of rare and very expensive isotope  $^{48}\text{Ca}$  is the main problem in attempts of the synthesis of superheavy elements.**

How to produce the beams?

Choose a proper working substance ( chemical form of element to be used as a projectile) and a technique of feeding an ECR ion source with it.

One needs working substances with:

1. high atomic content of the element in molecule,
2. relatively high volatility at moderate temperatures for a fine adjustment of the evaporation rate,
3. absence in the molecule of working substance of elements littering an ECR ion source and disturbing its work.

## **Evaporation techniques to feed an ECR ion sources with working substances**

1. **Ion sputtering** (negatively biased metallic sample is placed close to the ECR plasma so that positive ions from the plasma are accelerated toward the sample striking its surface and metal atoms are evaporated).

2. **Laser ablation** (laser evaporation of solid material in an ion source)

3. **Plasma heating** ( a rod of metal or oxide is inserted into the plasma where energetic plasma electrons vaporize the sample).

4. **Oven heating** ( a metal or chemical compound is evaporated from a crucible).

5. **MIVOC** ( Metal Ion from Volatile Compounds) – sublimation or vaporation of organometallic or other volatile compounds ( with vapour pressure  $> 0.1$  Pa) at room temperature.

6. **Evaporation through a reaction with a plasma support gas** (refractory metal or oxide reacts with reactive plasma support gas : for example,  $\text{SF}_6$  reacts with a metal yielding volatile fluorides).

**MIVOC** ( Metal Ion from Volatile Compounds) – sublimation or vaporation of organometallic or other volatile compounds ( with vapor pressure  $> 0.1$  Pa ) at room temperature;

- + low consumption rate (expensive isotopes may be used),
- + the evaporation is independent of the plasma characteristics and can be tuned finely,
- + the technique allows to reach high currents of metal ions,
- + easy way to feed an ion source,
- carbon contamination of a source,
- difficult to optimize the charge state distribution with support gas because of a large amount of C and H ions.

BUT

1. Calciocenes are polymeric solids.
2. Volatile calcium compounds used for a CVD technique have very low calcium ratio in molecule ( $\text{Ca(hfa)}_2 \times \text{tetraglyme}$ , where hfa – 1,1,1,5,5,5-hexafluoro-2,4-pentanedione, has a ratio of 1:65).
3. Very low yields in syntheses.

### **Oven heating**

- + low consumption rate (expensive isotopes may be used),
- + the evaporation is independent of the plasma characteristics and can be tuned finely,
- + the technique allows to reach high currents of metal ions,
- + different kinds of chemical compounds can be used for evaporation
- only temperature below  $1550$  °C is available.

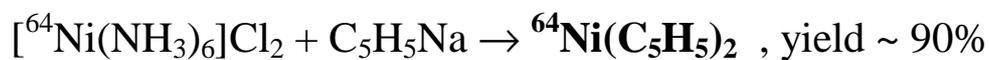
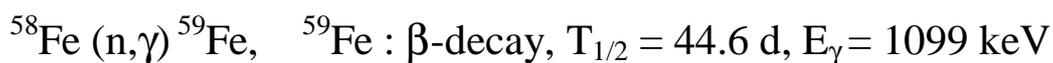
These considerations and our experiments have showed, that the best way of producing highly intense calcium ion beams is evaporation of metallic calcium by oven heating in an ECR ion source.

**MIVOC**  
**(Metal Ions from Volatile Compounds)**  
**for  $^{58}\text{Fe}$  and  $^{64}\text{Ni}$**

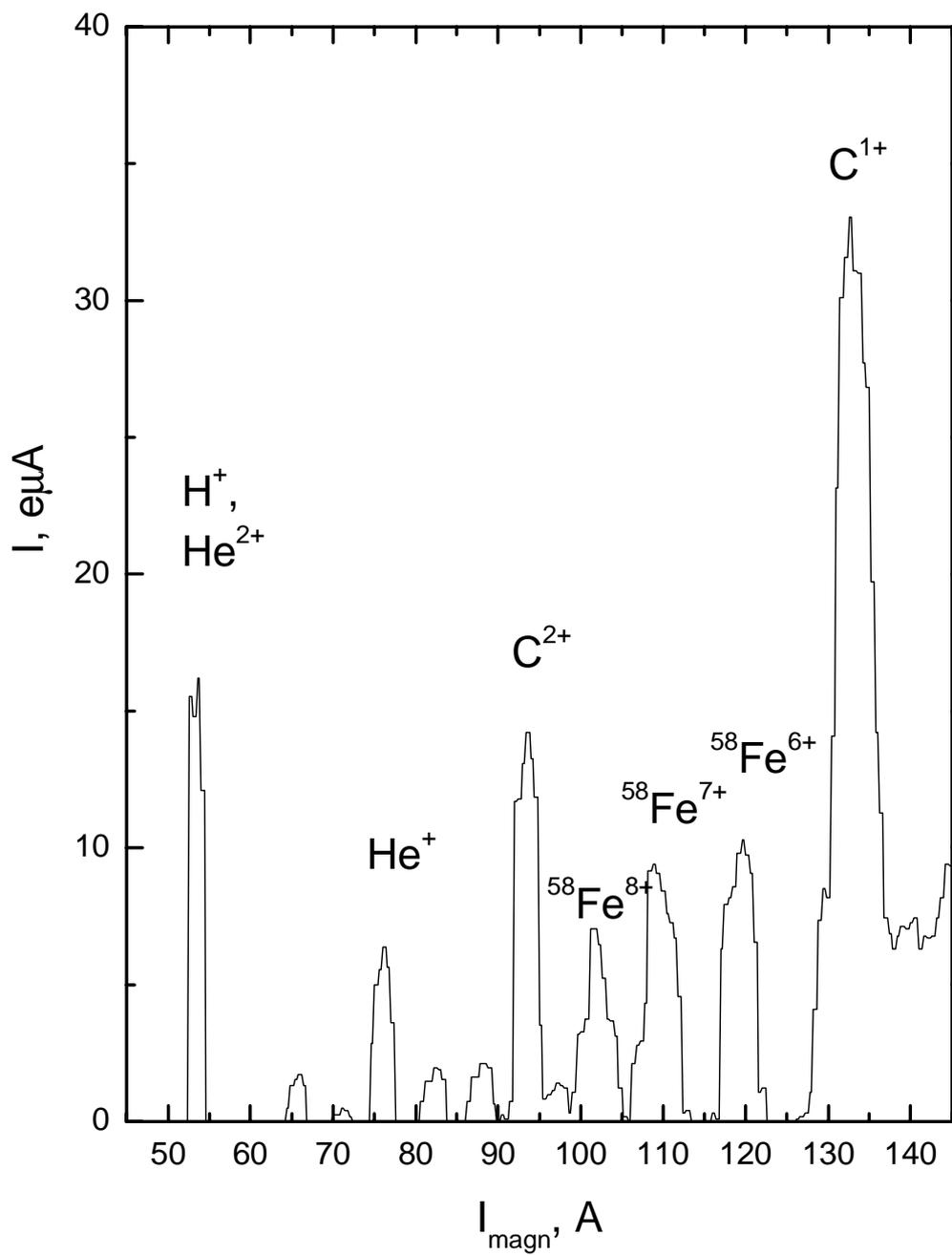


Vapour pressure (20°C) –  $1.7 \times 10^{-3}$  mbar

Radiotracer control

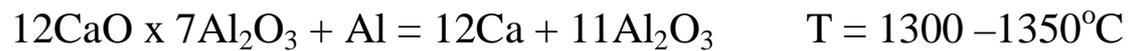


Vapour pressure (20°C) –  $3.5 \times 10^{-3}$  mbar



## **Production of metallic $^{48}\text{Ca}$**

Mixture  $^{48}\text{CaO} + \text{Al}$  (50-70% excess of Al)



Yield – 50-65%, production rate – 10-20 mg/h

## **Radiotracer control**

CaO + Al mixture was irradiated with  $E_e = 22 \text{ MeV}$   
bremsstrahlung



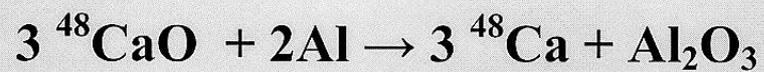
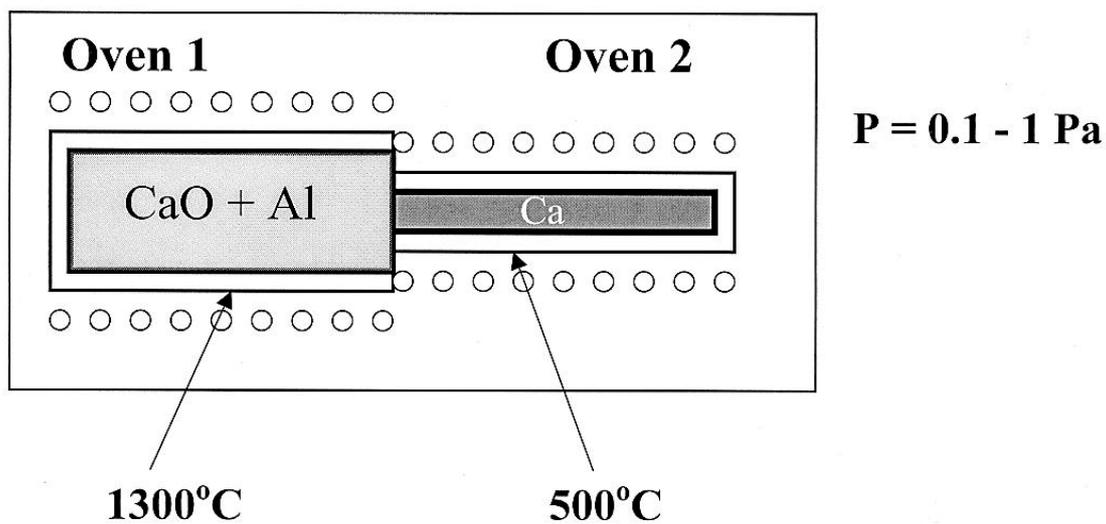
## **Production of metallic $^{26}\text{Mg}$**

Mixture of  $^{26}\text{MgO} + \text{Al}$



Yield – 90%

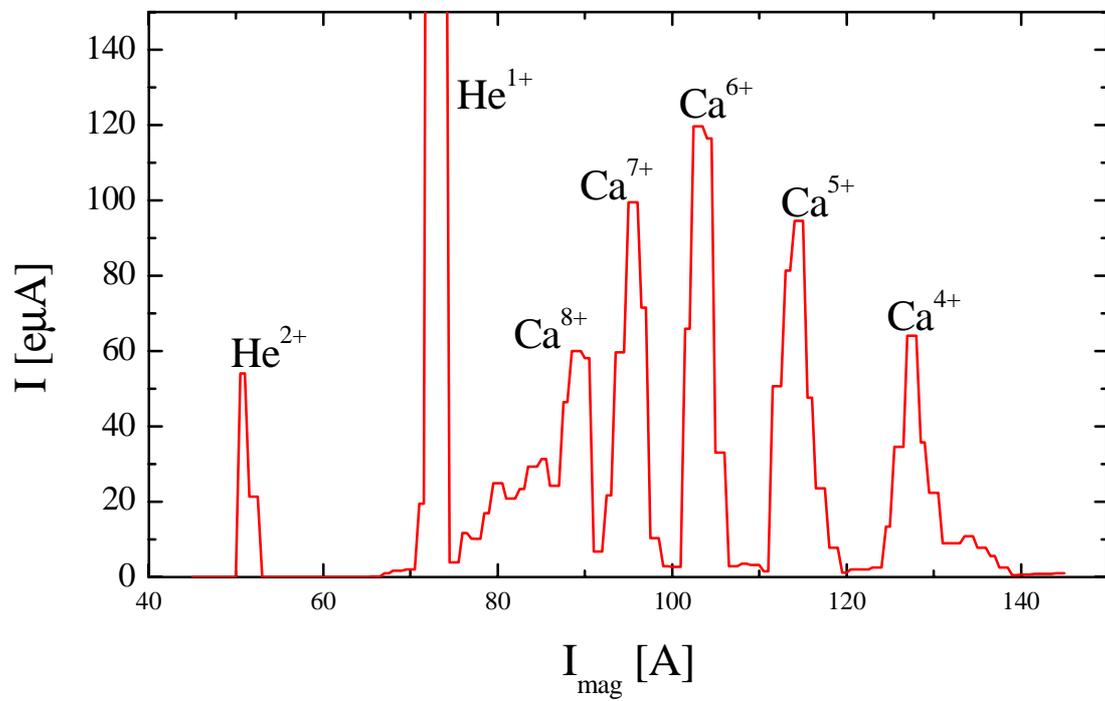
# PRODUCTION of METALLIC $^{48}\text{Ca}$



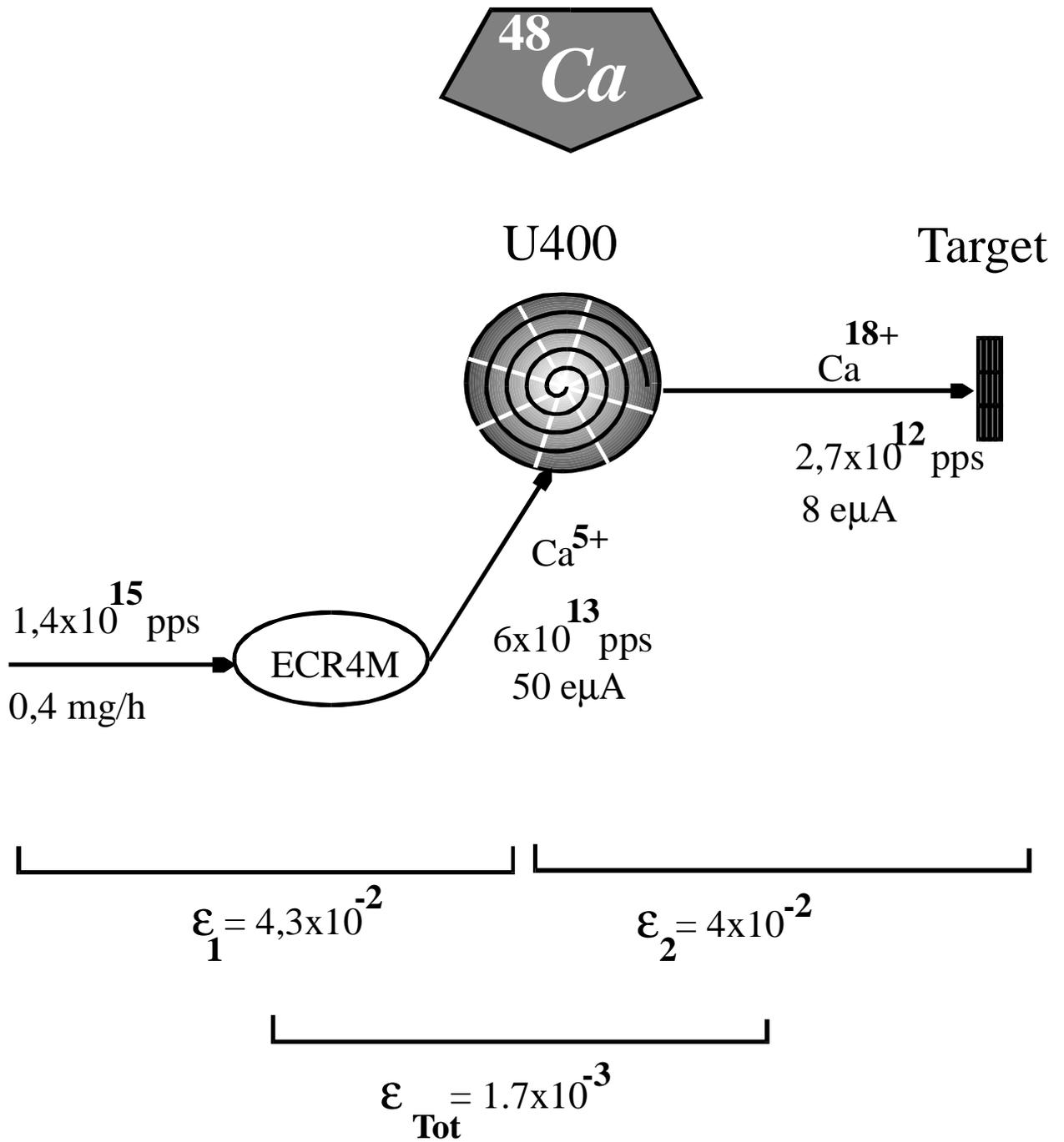
## Scheme of $^{48}\text{Ca}$ recuperation

1. Dissolving of a  $12\text{CaO} \times 7\text{Al}_2\text{O}_3$  residue from the  $^{48}\text{Ca}$  reduction process in conc. HCl acid.
2. Filtration and evaporation of HCl solution up to 10-15 ml.
3. Dilution of the solution to adjust calcium concentration of  $\sim 0.5$  g/l.
4. Addition to the boiling solution diluted ammonia solution (1:4) till pH = 7.
5. Boiling of a precipitate in the maternal solution during 2 min., filtration on a paper filter and washing of the precipitate with hot 2%  $\text{NH}_4\text{Cl}$  solution.
6. Addition to the boiling filtrate 4% ammonium oxalate solution and boiling during 2 min.
7. Filtration of a calcium oxalate on a track membrane after cooling of the solution, drying of a precipitate at  $90\text{-}100^\circ\text{C}$ .
8. Incineration of the calcium oxalate at  $1100^\circ\text{C}$  to produce  $^{48}\text{CaO}$  for the reduction process.

Fig.6 The spectrum of  $^{48}\text{Ca}$  ions optimised for production of  $^{48}\text{Ca}^{5+}$



EFFICIENCY of the ECR4M + U400



$1 \text{ g} / 0,0004 \text{ g} \times \text{h}^{-1} = 2500 \text{ hs}$

## Synthesis of superheavy elements

Nuclear reactions	At a target Beam intensity,pps	Beam dose	Consumption rate, mg/h
$^{238}\text{U} (^{48}\text{Ca}, 3n) ^{283}112$	$2 \times 10^{12}$	$3.5 \times 10^{18}$	0.4
$^{242}\text{Pu} (^{48}\text{Ca}, 3n) ^{287}114$	$4 \times 10^{12}$	$7.5 \times 10^{18}$	0.5
$^{244}\text{Pu} (^{48}\text{Ca}, 4n) ^{288}114$	$4 \times 10^{12}$	$1 \times 10^{19}$	0.5
$^{244}\text{Pu} (^{48}\text{Ca}, 3n) ^{289}114$	$4 \times 10^{12}$	$5.2 \times 10^{18}$	0.5
$^{248}\text{Cm} (^{48}\text{Ca}, 4n) ^{292}116$	$6 \times 10^{12}$	$8 \times 10^{18}$	0.75

### In future

1. Ion current increasing up to  $9 \times 10^{12}$  pps due to elimination of the external channel of injection of the ECR-4M.

2. Target thickness increasing up to  $1 \text{ mg/cm}^2$ .

It will be possible to synthesize 1 atom of element 114 per 3-4 days, so that investigations of its chemical properties become real.

## Conversion of SF<sub>6</sub> to other compounds of sulphur

1. A packed bed in a thick-walled quartz tube (a reactor) was a ceramic material (crushed baked clay tubes) boiled in PdCl<sub>2</sub> solution and dried at 100°C.
2. Dry H<sub>2</sub> was transported through the reactor and reacted at room temperature with PdCl<sub>2</sub> · 2H<sub>2</sub>O on a ceramic surface yielding a thin layer of palladium as a catalyst.
3. A mixture of SF<sub>6</sub> + H<sub>2</sub> (5-to 10-fold excess) reacted at 700°C over the catalyst in the reactor: SF<sub>6</sub> + 4H<sub>2</sub> = H<sub>2</sub>S + 6HF
4. HF was absorbed in a water scrubber made of teflon.
5. H<sub>2</sub>S reacted with J<sub>2</sub> in KJ<sub>3</sub> scrubbers: H<sub>2</sub>S + KJ<sub>3</sub> = S + 2HJ + KJ
6. Unreacted SF<sub>6</sub> (< 10%) was frozen in a liquid nitrogen trap to be used again in the conversion reaction.
7. Sulphur was washed by a hot KJ solution to remove the iodine impurity and was recrystallized in benzene.
8. Sulphur was burnt in a quartz tube in the mixture of dry oxygen and helium and resulting SO<sub>2</sub> was collected in U-tube cooled by vapour of liquid nitrogen.  
About 200 L of SF<sub>6</sub> enriched in <sup>34</sup>S (80-99%) was converted to S and SO<sub>2</sub> with small losses. The technique may be applied to SF<sub>6</sub> enriched in very expensive <sup>36</sup>S.



## Conversion of SF<sub>6</sub>

