MoO_3 PURIFICATION FOR SEARCHING ON NEUTRINOLESS DOUBLE BETA DECAY FOR AMORE

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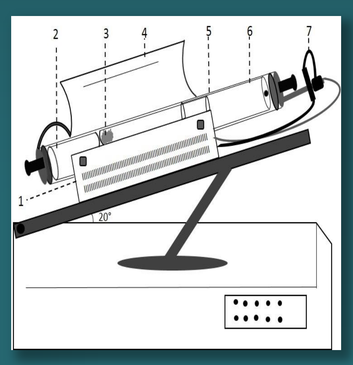
Motivation and introduction

- The AMoRE (Advance Molybdenum Based Rare process Experiment) collaboration is using scintillating crystals containing molybdenum to search for neutrionoless double decay of ^{100}Mo.
- The sensitivity of the detector used by AMoRE is determined by the internal background in the region of expected peak.
- To reduce the internal background from scintillating crystal the radioactive isotopes, which contribute around 3.03 MeV (^{208}Tl from the ^{232}Th chain and ^{214}Bi among the ^{226}Ra decay products in the ^{238}U chain), must be removed from the raw materials.
- The objectives of present study is to develop the best purification method for MoO_3 powder along with high yield efficiency.
- Different purification techniques such as: Sublimation, Recrystallization and Co-precipitation are studied for removing radioactive contaminants.
- The effectiveness of the purification techniques are checked with ICP-MS measurements and radioactivity from Ra, Th and U with HPGe detector at YangYang underground laboratory in Korea.

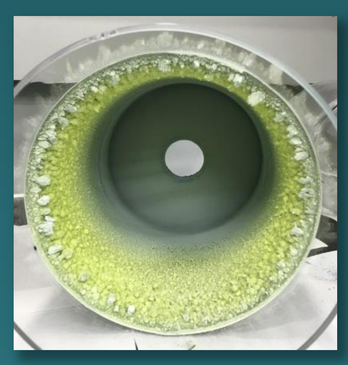
Experimental Procedure

Purification by sublimation

- Loading of MoO_3 powder (99.95%) in quartz tube.
- Loading quartz tube in sublimation machine.
- Operating the machine at 720 ˚C and vacuum condition (<10 mtorr)



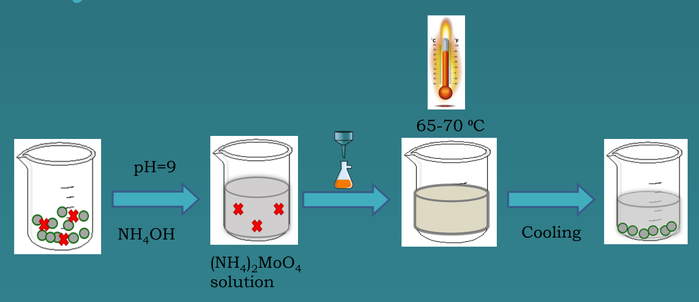
Schematic diagram of the experimental apparatus for MoO3 sublimation:
(1) Heater; (2) Left quartz tube; (3) Sample in middle quartz tube; (4) Thermal cap;
(5) Outer quartz tube; (6) Right quartz tube; (7) Nitrogen gas supply



MoO_3 powder after sublimation

Purification by recrystallization

- MoO_3 dissolved in NH_4OH
- Evaporation at the temperature of 65-70 ℃.
- Cooling of the solution at room temperature.
- Obtained product is PolyAmmonium Molybdate (PAM)

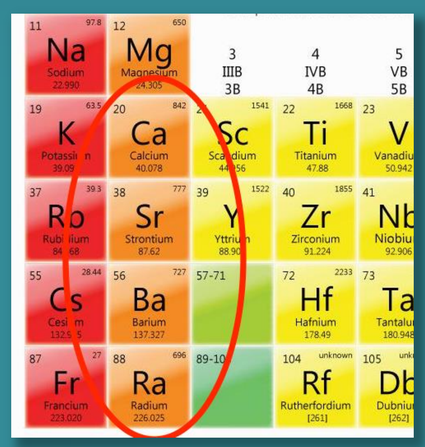


$$MoO_3 + 2\,NH_4OH \rightarrow (NH_4)_2MoO_4 + H_2O$$

$$7(NH_4)_2MoO_4 + 4H_2O \rightarrow (NH_4)_6Mo_7O_{24}.4H_2O + 8NH_4OH$$

Purification by co-precipitation

- Leaching MoO_3 in NH_4OH.
- Adding carrier $CaCl_2$ at pH 9 to precipitate 3% Mo.
- Exposition of carrier in the solution for long time to occur post-precipitation.
- Separation of $CaMoO_4$ precipitate through filtration

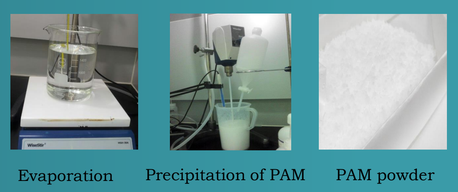


$$(NH_4)_2MoO_4 + H_2O + CaCl_2 \rightarrow CaMoO_4\downarrow + Ca(OH)_2\downarrow + (NH_4)_2MoO_4 + NH_4Cl$$

Obtaining of final product

Final product was obtained by two ways:

1. Complete evaporation of solution at 70 ˚C.
2. Precipitation of PAM by adding HCl.



Evaporation | Precipitation of PAM | PAM powder

$$(NH_4)_2MoO_4 + H_2O + HCl \rightarrow (NH_4)_2Mo_4O_{13}.2H_2O + NH_4Cl$$

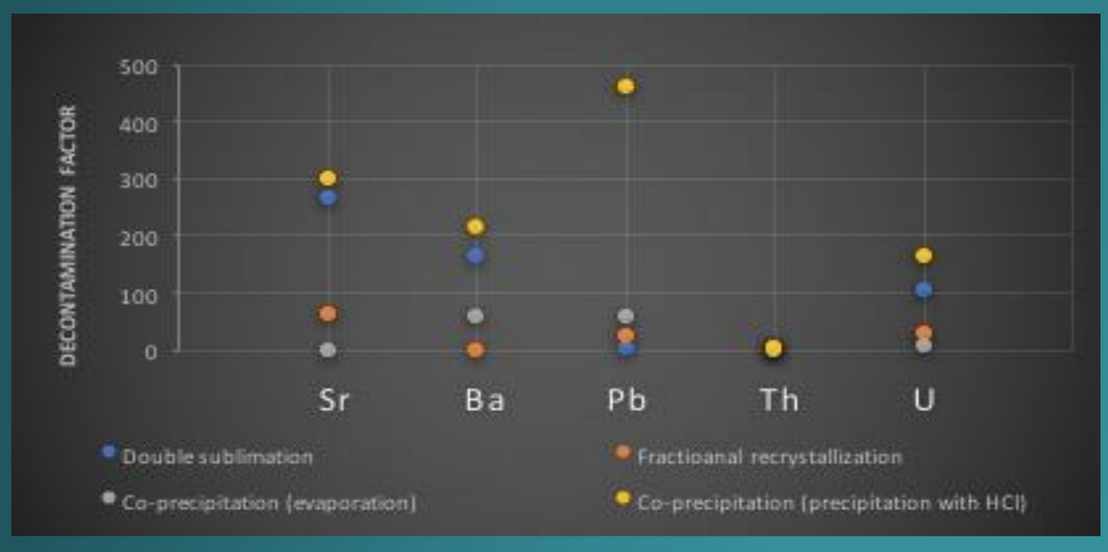
Results and Discussion

Effectiveness of MoO_3 purification by sublimation

Description	Sr, ppt	Ba, ppt	Pb, ppt	Th, ppt	U, ppt
Initial powder	23714	2829035	107027	91	7014
Single sublimation	930	123076	68853	<50	612
Decontamination Factor	25	23	2	>2	11
Double sublimation	89	17084	60296	<35	66
Decontamination Factor	266	166	2	>2	106

Effectiveness of MoO_3 purification by wet chemistry and sublimation

Elements	Initial Impurities (ppt)	Decontamination factors (DF)			
		Double sublimation	Fractional recrystallization	Co-precipitation (evaporation)	Co-precipitation (precipitation with HCl)
Sr	23714	266	61	0.5	301
Ba	2829035	166	0.57	58	215
Pb	107027	2	23	60	462
Th	91	≥2	≥2	≥0.63	≥3
U	7014	106	27	6	≥164



Samples	Ba	^{232}Th	^{238}U	^{226}Ra (U)	^{228}Ac (Th)	^{40}K
		ppt			mBq/kg	
$^{100}MoO_3$ (99.997%)	16,600	< 46	73	8.3	< 1	9
	11,400	< 61	149	3.8	< 0.8	36
Ini. MoO_3	2,829,035	91	7,014	1,627	851	725.48
Single Sub.	123,076	< 50	611.8	124.7	49.9	73.84
Double sub.	17,084	< 35	66.09	20.56	6.73	74.80

- The most effective purification technique is co-precipitation with $CaCl_2$ as a carrier and successive precipitation with HCl from acidic media. Next one is double sublimation which is characterized high efficiency above 99%. Combination of sublimation and wet chemistry technique is very promising way to achieve purity level below enriched $^{100}MoO_3$ powder