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Production of radiopure natural and isotopically enriched cadmium and zinc for low background scintillators

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A new approach to deep purification of natural and isotope-enriched cadmium and natural zinc to grow high-quality radio-pure crystal scintillators has been developed. Purification of Cd and Zn by distillation through getter filters has been investigated. Application of the complex refinement method (heating with filtration in combination with distillation through getter filter) provides the best efficiency of purification. The developed method allows to achieve high purification efficiency, high yield of product and low losses of the initial metals.

Представлены результаты разработки новых приемов глубокого рафинирования природных и изотопно обогащенных кадмия и природного цинка для выращивания качественных и радиоактивно чистых сцинтилляционных кристаллов. Исследован процесс дистилляции Cd и Zn через геттерные фильтры. Показано, что применение комплексного метода рафинирования (прогрев с фильтрацией в сочетании с дистилляцией через геттерный фильтр) обеспечивает получение высокочистых материалов с высокой эффективностью очистки, высоким выходом годного продукта и низкой потерей исходного металла.

1. Introduction

Crystal scintillators are promising detectors for high sensitivity double beta decay experiments [1, 2]. Several 2β active isotopes can be investigated using crystal scintillators containing cadmium and zinc: $CdWO_4$ (^{106}Cd, ^{108}Cd, ^{114}Cd, ^{116}Cd), $ZnWO_4$ (^{64}Zn, ^{70}Zn, ^{180}W, ^{186}W), $ZnMoO_4$ (^{64}Zn, ^{70}Zn, ^{92}Mo, ^{98}Mo, ^{100}Mo), ZnSe (^{64}Zn, ^{70}Zn, ^{82}Se).

Search for dark matter can be realized using crystal scintillators as cryogenic scintillating bolometers. For instance, the EURECA project aims to use different crystal scintillators to verify nature of dark matter signal: $ZnWO_4$, $CaWO_4$, $CaMoO_4$, $MgWO_4$, CaF_2, BGO, Al_2O_3, LiF, $PbWO_4$, $PbWO_4$, $PbMoO_4$, etc. The EURECA dark matter project requires high light yield of scintillators (>15.000 photons/MeV) which can be achieved provided a high quality of crystals. Suppression of detector background counting rate to the level less than a few events per keV/100 kg/year in the energy interval of a few keV is a critical issue for success of the experiment. To achieve such an extremely low background, crystal scintillators with radiopurity on the level of <0.01 mBq/kg are to be developed [3]. Similarly, the radiopurity level of crys-

tals for future 2β decay experiments should not exceeds a few μBq/kg for ^{228}Th and ^{226}Ra. For instance, $ZnWO_4$ is a good example of radiopure scintillator (total α activity ~0.2–1 mBq/kg level [4]). However, at least 20-fold improvement of $ZnWO_4$ radiopurity is still needed for the EURECA experiment.

The main requirements to the content of chemical impurities in initial metals and raw materials for high-quality optical quality scintillators are as follows: Fe, Mg, Mn, Cr, V, Co < 1 ppm; Ni, Cu < 0.2 ppm. Obviously, contamination with radioactive elements: K, Rb, Lu, La, Sm, U, Th should be as low as possible, too. To achieve such a level of metals purity, new advanced refining techniques should be developed.

One of the main procedures to produce high-purity cadmium and zinc is vacuum distillation. There is a known method of refinement by vacuum distillation with a condensation under temperature gradient on the condenser that is accompanied by the condensing metal re-evaporation [5]. A disadvantage of this method is its low efficiency because condensate of purified metal is spread out on the condenser along the temperature gradient, which requires mechanical separation of the most pure part of the condensate.

A much more effective method is the two-stage distillation by using the distillation device described in the patent [6]. The first stage includes the distillation of volatile impurities and removing oxide layer from the surface of melted metal. The metal heated up to the melting temperature flows down into the crucible through a hole in a plate. During this procedure, volatile (Se, K, Rb, Cs, Fr, S, P) impurities and volatile oxides of heavy metals are condensed in a main condenser together with about 5 % of the metal being refined. Heavy oxides of impurity metals and slags remain on the plate surface in the form of a thin film. During the second stage, after changing the main condenser for the additional one, the metal can be purified from heavy volatile impurities (Cu, Fe, Ni, Co, Ag, Pb, Tl, Sb, Bi, Li, Sn, Mn, Ra, Th, U etc.) by additional distillation. During this stage, approximately 5–10 % of initial metal remains in the crucible. The condensate obtained at this stage is the final refined product. Recently, a new purification method for Cd, Zn and Te from interstitial impurities by vacuum distillation using getter filters was proposed [7, 8]. Effectiveness of getter filters made of the alloy Zr(51 wt.%)–Fe(49 wt.%) was demonstrated for purification of zinc from gaseous impurities and carbon: the content of these impurities in the purified metal is one order of magnitude lower than that in the initial metal. At the same time, the content of N, C, O in the distillated metals without the getter have been decreased only by 2–3 times. Analysis of metal samples contamination has shown that application of a Zr–Fe getter filter for Zn distillation provides additional purification by a factor of 2–5. This result was used for further investigations to purify natural and isotope-enriched cadmium and natural zinc.

The aim of this work is to research and develop the deep purification methods for cadmium and zinc by heating with filtration and subsequent distillation through getter filters. Selection of available and inexpensive materials for the getter filters was an important part of the present study.

2. Selection of getter materials

Getter filters are commonly used for noble gases purification. Non-evaporable getters based on Zr, Ti alloys and compounds (known as NEG materials) are widely applied in modern technologies to obtain high purity gases. The most investigated and extensively used are the Zr–Fe, Zr–Al, Zr–V, Zr–Ni and Ti–V alloys; ternary alloys Zr–Mn–Fe, Zr–V–Fe, Ti–V–Mn; multicomponent alloys Zr–Ni–A–M or Zr–Co–A, where A are the elements like yttrium, lanthanum, rare-earth metals or their compositions; M can be cobalt, copper, iron, aluminum, tin, silicon or their compositions. Compositions of the above-mentioned metals and alloys can be used, too [9].

Getter properties of chemically active metals, intermetallic compounds and alloys on the base of zirconium and hafnium were investigated in the NSC KIPT [10]. These materials, operating in the temperature region of 200–500°C, can be reused after the appropriate activation. According to [10], alloys of zirconium and hafnium with iron possess high sorption ability. One of such alloys, Zr(51 wt.%)–Fe(49 wt.%), has been used to purify zinc and cadmium.

Getter materials for purification of metals should satisfy the following requirements:

— high chemical activity relatively to gas impurities (H, N, O) and carbon containing gases;

— as low as possible reactivity with vapors of the purified metal;

— low vapor tension values at the operation temperature;

— high sorption activity at the distillation process temperatures;

— activation temperature providing the minimum duration of the activation process.

Gases (N, CH_4, CO, O_2) are adsorbed on chemically active getter surface at 300–500°C with subsequent diffusion into the getters at higher activation temperatures. The processes of metal purification during distillation through getter filter have been shown to differ substantially from the mechanism of gases purification. In our opinion, when the vapor of the initial metal passes though the filter, the impurity elements are adsorbed on the filter due to the multiple re-evaporation of the initial metal on the filter material.

Because gaseous contamination is not so crucial for crystal scintillators production, we have investigated, in addition to Zr–Fe getters, more available and cheaper materials for the getter filter: shavings of high-purity zirconium and shards of high-purity graphite.

3. Behavior of impurity elements in cadmium and zinc during distillation

Cadmium and zinc are the neighboring elements in the II group of the Periodic Table of elements and have similar physical-chemical properties (crystal structure, melting temperatures, vapor tension, interaction with materials in liquid condition, etc).

Distillation method of metal purification is based on difference in the compositions of melt and vapor. The difference can be expressed through relative volatility (separation factor α) of the components to be separated. In a case of an ideal diluted solution and conditions of molecular evaporation, factors α_i can be calculated according to [11]. The calculated values of factors α_i for zinc and cadmium in the temperature range from 600 to 900 K are given in [12, 13].

Analysis of the α_i values for Cd and Zn in the temperature range from the melting point to the boiling point has shown that the spectrum of impurity elements includes volatile impurities Se, K, Rb, Cs, Fr, S, P ($\alpha_i < 1$), heavy-volatile impurities Fe, Mg, Mn, Cr, V, Co, Ni, Cu, Bi, Pb, Ra, Th, U ($\alpha_i > 1$) and several elements: As, Te, Na, with $\alpha_i \sim 1$. Separation of the latter elements is much difficult. At the same time, the transition and radioactive elements most dangerous for scintillation crystals can be removed effectively: calculated values of α_i exceed 10^2.

The behavior of impurity elements in Cd and Zn during distillation process was considered in [12, 13]. Investigations of the efficiency of Cd and Zn purification from volatile elements depending on the degree of distillation at a given temperature were carried out. More than 10-fold purification of the melt from volatile impurities (with $\alpha_i < 1$) can be reached under condition of <10 % evaporation of Cd and Zn. For the heavy impurities (with $\alpha_i > 1$) more than 10-fold refinement can occur if more than 95 % of Cd and Zn become to condensate.

Therefore, deep purification of cadmium and zinc can be reached in step-by-step purification by using getters with loss of the main material at the level of 5–10 %. We have proposed to use getter filter at the stage of purification from heavy-volatile impurities. Besides, the combination of prefiltration and distillation using the getter filter with subsequent molding of distillates into the ingots in the casting unit was chosen to minimize losses of isotope-enriched cadmium instead of a step-by-step purification procedure.

To develop a distillation apparatus with getter filter, as well as to choice the optimal temperature and time regimes of distillation, calculation of purification efficiency were carried out.

4. Process of natural zinc and isotope-enriched cadmium refinement by vacuum distillation through getter filter

The initial materials to be refined were granular Cd of analytically pure grade (spec 6-09-3095-78), Zn ingots of ZV00 grade (State Standard 3640-94), samples of cadmium enriched in ^{106}Cd (68 %) and ^{116}Cd (83 %).

High-purity accessory materials and equipment were used to minimize possible contamination of the materials in the course of purification procedure. The crucible and the condenser of distillation apparatus were made of high-purity graphite of MPG-7 grade. Details of the heater and screens were made of the spectrally pure graphite, corresponding to specification 48-20-90-82. The content of controlled impurities in such graphite is $\leq 6.2 \cdot 10^{-4}$ wt.%. Argon gas of 99.995 % purity grade (the State Standard

10157-88) was used as an inert atmosphere for Cd and Zn filtration procedure.

Contamination of the initial and purified cadmium samples was analyzed by ICP-MS-Inductively coupled plasma mass-spectrometry (Laboratori Nazionali del Gran Sasso of INFN, Italy), LMS-Laser mass-spectrometer (NSC, KIPT, Kharkiv, Ukraine), AAS-Atomic absorption spectroscopy (LNGS of INFN, Italy). The AAS method was applied to determine the iron concentration in cadmium (the sensitivity of ICP-MS was too low due to interference of iron isotopes with ^{58}Ni and doubly ionized ^{112}Cd and ^{114}Cd).

Quantitative analysis of zinc samples was performed by SMS-Spark mass-spectrometry (Giredmet, Moscow, Russia), LMS-Laser mass-spectrometry (NSC, KIPT), Inversion volt-amperometric method (STC "Institute for Single Crystals", Kharkiv, Ukraine).

The accuracy of the analytical methods was estimated to be at the level of 15–30 %.

5. Experimental technique, results and discussion

The efficiency of cadmium and zinc purification can be improved by using getter filters in distillation process. In this method, the vapors of the metal to be refined are passed through the filter made of a getter material heated to the boiling temperature of the refined metal. The scheme of Cd and Zn refinement in vacuum using the getter filter is presented in Fig. 1.

The filter material as shards or shavings is placed on a mesh in a crucible closely above the melted metal inside the quasi-closed distillation apparatus. The following materials were used as getters: shards of Zr–Fe alloy, shavings of high-purity Zr, and shards of high-purity graphite. The thickness of the getter was about 1 cm.

The gaseous impurities released during distillation are absorbed by the getter, thus causing a decrease of content thereof in the condensate. Some additional purification occurs due to dissociation of volatile metal oxides with subsequent absorption of the elements at the getter surface due to the increased activity of the getter to oxygen. However, the main mechanism of the additional purification on the getter is the multiple repeated evaporation of the metal being refined during its passing through the getter material. Graphite, being available and inexpensive, is a good getter material to purify Cd and Zn from the metal impurities. At the same time, filter does not absorb the material to be purified because it is heated to the evaporation temperature of the purified metal.

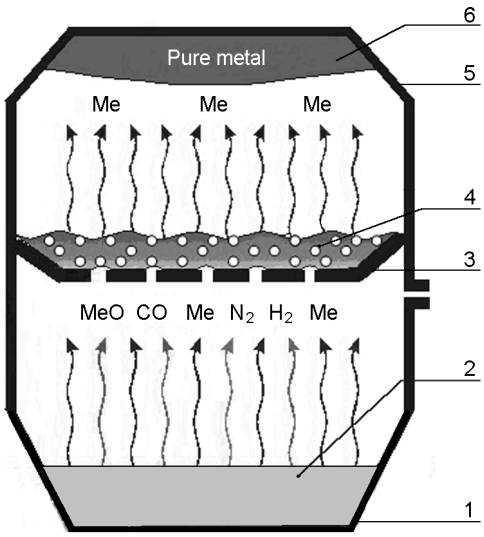

Fig. 1. Schematic view of cadmium and zinc purification in vacuum using getter filter: *1*, crucible; *2*, initial metal; *3*, cellular plate; *4*, getter; *5*, condenser; *6*, purified metal.

We have revealed 10-to 100-fold purification from metal impurities (Ni, Cu, Fe, Mg, Mn, Cr, V, Co, Th, U, Ra, K, Rb, Bi, Pb, Lu, Sm) in Cd and Zn without filter, and 20-to 500-fold purification when using filters. Reduction of interstitial impurities (N, C, O) in Cd and Zn is by 3 times without filter, while application of Zr–Fe provides 20-fold refinement.

The method described above resulted from the modification of the distillation apparatus described in [8, 14]. Summary of Cd and Zn contamination before and after purification is given in Table 1.

As is seen from Table 1, the obtained Zn is of higher purity than the Cd. This result can be explained by higher purity of the initial material and the application of a getter filter.

Table 2 presents the impurity content of isotope-enriched Cd prior to and after purification. The purification efficiency using the graphite filter is twice higher than that after refining by only distillation. According to the LMS analysis, the purified ^{116}Cd has lower contamination in comparison to ^{106}Cd. To confirm this results, the contamination is to be measured by more sensitive methods, for example, by ICP-MS.

Data on microhardness (H_μ) of natural Cd and enriched ^{116}Cd after refinement by distillation using different filters are presented in Table 3. The microhardness was measuted using a PMT-3 instrument (load

Table 1. Contamination of natural Cd and Zn prior to and after refining. The sign ≤ indicates detection limits of analytical methods: for Cd, *ICP-MS, **LMS, ***AAS; for Zn, *SMS, **LMS, ***Inverse volt amperometric method

Impurity Element	Concentration in Cd (ppm)		Concentration in Zn (ppm)	
	prior to	after refining	prior to	after refining (with Zr–Fe getter)
Ni	30*	0.3*/≤0.2**	0.3**	< 0.04*/≤0.03**/<0.03***
Cu	47*	0.3*/≤0.2**	10**	<0.1*/≤0.04**/0.3***
Fe	0.4***	0.17***/≤0.5**	30**	0.1*/≤0.08**
Mg	30*	≤0.5*/≤0.05**	0.7**	<0.02*/≤0.08**
Mn	0.2*	0.1*/≤0.3**	<0.2**	<0.04*/≤0.02**
Cr	0.2*	0.1*/≤1**	<0.2**	<0.05*/≤0.02**
V	<0.005*	≤0.005*/≤0.08**	<0.2**	<0.04*/≤0.02**/<0.05***
Co	0.3*	≤0.003*/≤1**	<0.2**	<0.05*/≤0.02**
K	8*	≤5*/0.7**	3**	<0.05*/0.05**
Pb	1000*	3*/≤1**	30**	<0.6*/≤0.3**/<0.01***
Th	<0.001*	≤0.001*/<0.2**	<0.2**	<0.2*/≤0.2**
U	<0.001*	≤0.001*/<0.2**	<0.2**	<0.2*/≤0.2**

Table 2. Impurity content in enriched ^{106}Cd and ^{116}Cd prior to and after refining

Impurity elements	Concentration in ^{106}Cd (ppm) [15]		Concentration in ^{116}Cd (ppm)		
	prior to refining	after refining	prior to refining	after refining	
		4-fold distillation		4-fold distillation	double distil. with graphite filter
Ni	0.6*	0.6*/≤0.2**	≤0.2**	≤0.2**	≤0.2**
Cu	5*	0.7*/0.5**	0.7**	≤0.1**	≤0.1**
Fe	1.3***	0.4***/≤0.4**	≤5**	≤5**	≤5**
Mg	12*	≤0.5*/≤0.05**	≤0.05**	≤0.05**	≤0.05**
Mn	0.1*	0.1*/≤5**	≤5**	≤5**	≤5**
Cr	9*	≤0.5*/≤0.1**	≤0.1**	≤0.1**	≤0.1**
V	<0.005*	≤0.01*/≤0.08**	≤0.08**	≤0.08**	≤0.08**
Co	0.02*	≤0.01*/≤0.1**	≤0.1**	≤0.1**	≤0.1**
K	11*	≤10*/0.04**	5.4**	≤0.04**	≤0.04**
Pb	270*	8*/≤0.3**	80**	≤0.7**	≤0.7**
Th	<0.001*	<0.001*/≤0.6**	≤0.6**	≤0.6**	≤0.6**
U	<0.001*	<0.001*/≤0.6**	≤0.6**	≤0.6**	≤0.6**

*ICP-MS, **LMS, ***AAS — Atomic Absorption Spectroscopy.

20 g) by indentation of a diamond pyramid in a metal sample. Values of $H\mu$ for Cd samples after refinement are lower than the reference value. This is an indirect evidence of the higher purity of Cd after refinement. Value $H\mu$ = 28.6 kg/mm^2 for isotopically enriched ^{116}Cd was obtained for the first time.

The high-purity ingots of natural cadmium (463 g) and zinc (950 g), enriched ^{106}Cd (152 g) and ^{116}Cd (252 g) have been obtained by using the developed complex refinement method (heating with filtration in combination with distillation through getter filter). This method allows to achieve purified metal concentration exceeding 99.999 %, high yield (>96 %), and low irrecoverable losses (<1 %) of the initial metals.

Table 3. Microhardness of natural Cd and enriched ^{116}Cd after refining by distillation using different filters (kg/mm^2)

Reference value [16]	# 1, Cd without filter	# 2, Cd Zr filter	# 3, Cd Zr–Fe filter	# 4	Cd graphite filter
30.0	28.2	28.9	27.3	27.2	28.6

A cadmium tungstate crystal scintillator enriched in ^{106}Cd to 66 % ($^{106}CdWO_4$) was developed for a high sensitivity experiment to search for 2β processes in ^{106}Cd. The cadmium tungstate compounds (natural and enriched) to grow the $CdWO_4$ crystal scintillators were synthesized by the JSC Neo-Chem (Moscow, Russia) from the additionally purified solutions of cadmium nitrates and ammonium para-tungstate. The contamination of the compounds was measured by ICP-MS and ASS. The purity level of the powders satisfies the requirements to raw materials for cadmium tungstate crystals growth [17]. A single crystal of 231 g mass, that is 87.2 % of the initial charge mass, was grown from the enriched ^{106}Cd by the low-thermal-gradient Czochralski technique at the Nikolaev Institute of Inorganic Chemistry (Novosibirsk, Russia). The boule length was 60 mm, the cylindrical part diameter 27 mm. The crystal has practically neither coloration nor defects typical of $CdWO_4$ crystals. High optical and scintillation properties of the $^{106}CdWO_4$ crystal were reached thanks to the improved quality of the material as a result of the deep purification by the cadmium distillation procedure described in this work, the advanced chemistry applied to the powder synthesis, as well as thanks to the advantage of the low-thermal-gradient Czochralski technique for crystal growth [17]. The results demonstrate that $^{106}CdWO_4$ crystal scintillators have a good potential to search for double beta processes in ^{106}Cd. An experiment to search for double β decay of ^{106}Cd with the help of the developed $^{106}CdWO_4$ crystal scintillator is in progress in the DAMA R&D set-up at the Gran Sasso National Laboratories of the INFN (Italy).

As a next step, we are going to develop a large volume high-quality $^{116}CdWO_4$ crystal for a high sensitivity experiment to search for double beta processes in ^{116}Cd. The radio-pure high-quality $ZnWO_4$ crystal scintillators are expected to be developed for the EURECA cryogenic dark matter experiment.

6. Conclusions

A new method to purify cadmium and zinc (heating with filtration in combination with distillation through getter filter) has been developed. The method provides high purification efficiency, high yield of product and low losses of initial metals. Samples of Cd with the natural isotopic composition, enriched ^{106}Cd and ^{116}Cd, and Zn of high purity grade (≈99.999 %) have been obtained. The method can be used to purify cadmium and zinc for development of radiopure high-quality crystal scintillators for experiments to search for double beta decay and dark matter.

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Одержання радіоактивно чистих природних та ізотопно збагачених кадмію і цинку для низькофонових сцинтиляторів

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Представлено результати розробки нових методів глибокого рафінування природних та ізотопно збагачених кадмію та природного цинку для вирощування якісних та радіоактивно чистих сцинтиляційних кристалів. Досліджено процес дистиляції Cd та Zn через гетерні фільтри. Показано, що застосування комплексного методу рафінування (прогрів з фільтрацією у поєднанні з дистиляцією через гетерний фільтр) забезпечує отримання високочистих матеріалів з високою ефективністю очищення, високим виходом придатного продукту і низькою втратою вихідного металу.