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# Cadmium Purification by Comprehensive-Distillation Method

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An analysis of literature data for oxidative refining of cadmium is presented. A thermodynamic analysis of the oxidation reactions of Cd and the impurities Zn, Pb, Fe, Cu, Tl, Ni, Sn, As, Sb, *etc*. present in it during melting under standard conditions, as well as during distillation of cadmium in vacuum, is performed. The strength of oxide compounds of impurities in cadmium is evaluated by the absolute value of the change in Gibbs free energy in the condensed (liquid) state and in the vapour (vacuum) phase. A comprehensive method for Cd purification, including oxidizing refining and subsequent removal of non-volatile and volatile impurities in a single refining cycle is investigated.

**Key words:** cadmium, oxides of cadmium and impurities, Gibbs energy, oxidizing refining, filtration, distillation.

Представлено аналізу літературних даних стосовно окисного рафінування кадмію. Виконано термодинамічну аналізу реакцій окиснення Cd та присутніх у ньому домішок Zn, Pb, Fe, Cu, Tl, Ni, Sn, As, Sb та ін. за розтоплення у стандартних умовах, а також за дистиляції кадмію у вакуумі. Міцність окисних сполук домішок у кадмії оцінювали за абсолютною величиною зміни Ґіббсової вільної енергії у конденсованому (рідкому) стані та паровій (у вакуумі) фазі. Досліджено комплексний метод очистки Cd з окисним рафінуванням і наступним видаленням тяжко- та легколетких

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домішок в одному циклі рафінування.

**Ключові слова:** кадмій, оксиди Кадмію і домішок, Ґіббсова енергія, окисне рафінування, фільтрація, дистиляція.

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## 1. INTRODUCTION

High-purity cadmium (> 99.999 wt.%) is used in microelectronics, optoelectronics, and other fields of electronic engineering as a constituent component for the synthesis and growth of semiconductor compounds $Cd_3As_2$, $CdAs_2$, CdSb, CdTe. For that, various equipment and devices are manufactured [1]. The purity of the source elements determines the quality and reliability of such products.

Grown crystals of Cd chalcogenides are successfully used for the production of windows and lenses of continuous $CO_2$ lasers with a power up to 5 kW at the power density up to 100 kW/cm$^2$; various optical elements of devices within the visible and IR ranges (CdSe, CdS, CdTe); IR radiation polarisers (CdSe); oriented crystalline substrates for the creation of epitaxial structures (CdSe, CdTe) [2].

The important task is development of efficient and reliable solid-state semiconductor detectors for dosimetry of ionizing α- and γ-radiation in nuclear and future thermonuclear power. Semiconductor compounds CdTe and $Cd_{1-x}Zn_xTe$ (CZT) with a molar fraction $x = 0.1$–$0.4$ have advantages when used as the materials for the efficient ionizing radiation detectors owing to their unique combination of a number of electrophysical parameters.

Solid semiconductor solutions $Cd_xHg_{1-x}Te$ (CHT) having a molar fraction of $x = 0.2$–$0.3$ are principal materials for manufacturing photodetectors in the IR range (8–12 μm) [4]. As the requirements to epitaxial CHT structures increase, so do the demands to the purity of the source components, including gas impurities [4].

The synthesis of semiconductor compounds for microelectronics requires source elements having the purity > 5N, where the content of the electrically active majority impurities should be at $< 1\cdot10^{-5}$ wt.% [5–7]. Achieving this cadmium quality is not a trivial task and requires the development of special refining technological processes. Various aspects of this problem permanently attract the attention of researchers.

The developed Cd refining technologies are multistage and use a complex of methods [1, 8–15]. One of the radical means to increase the efficiency of metal purification is to successive use a number of refining methods that have different mechanisms for separating impurities. In this case, more efficient separation of impurities is expected than with repeated use of the single method, even a very effective that.

The increasing needs for high-purity cadmium and higher requirements for its purity determine the importance and relevance of currying out research to establish the regularities of behaviour of impurity elements in various refining methods and to develop effective processes for obtaining high-purity Cd. This is the subject of the research in the present work.

### 1.1. Relevance of the Problem and Formulation of the Task

From experiments on the melting of cadmium both under an inert gas atmosphere and in a vacuum, it is known that oxide films form on the surface of the molten metal. These films are formed as a result of chemical reactions of residual oxygen in the surrounding atmosphere with both the atoms of the base metal and the atoms of impurity elements.

To make useful application of the presence of oxide films on the surface of molten metal refined and to use the effect of their formation, the authors of a number of papers have described a technique such as filtration of liquid metal to remove oxide films. This approach can be conventionally classified as a method of oxidizing refining of metals. Oxidizing refining of metals is based on the difference in oxygen affinity between the base metal and impurities. If the impurity oxide is not soluble in the base metal, it floats to the surface and is removed from the crucible.

One of the options for oxidizing refining of cadmium is distillation in a stream of overheated water vapour [16]. The interaction of cadmium with water vapour can be called hydrothermal oxidation. Authors determined the coefficient of cadmium purification of zinc during its distillation with water vapour. During the distillation of cadmium, the temperature in the evaporation zone was maintained at about 920 K, and in the condensation zone at $\cong 590$ K. The total vapour pressure of cadmium and water was equal to atmospheric that.

The purification coefficient was calculated using the approximate formula: $\alpha = x/y$, where $x$ and $y$ are the impurity contents in the original and purified distillate, respectively. The results of experiments to determine the separation coefficient of zinc $\alpha$ during the distillation of cadmium with hydrothermal oxidation showed that the value of alpha in this case is equal to 235. Thus, during distilling cadmium with hydrothermal oxidation, it is possible to increase the efficiency of cadmium purification of zinc.

The issue of the effect of the presence of cadmium oxide on the degree of cadmium purification of impurities during the distillation process was considered in Ref. [14]. The study of the cadmium distillation process was carried out using quartz crucible of the horizontal type under the conditions of static and dynamic vacuum. The temperature of the evaporation zone was 820–870 K, the condensation temperature was 600–620 K. In all experiments, a loading with a mass of $G_0 \cong 1$ kg was used.

The content of impurities in cadmium at different stages of purification was monitored by the spectral method. The purification degree ($N_d$) was determined at the fraction of cadmium distillation $\varphi = 0.94$–$0.96$. $N_d = C_0/C_d$ is the ratio of the impurity content in the initial sample and the distillate. It was established that the presence of cadmium oxide, which is a collector of impurities, leads to an increase in the purification degree. This conclusion is confirmed by model experiments at the addition of cadmium oxide in the amount of 0.10–0.12% of the initial substance loading $G_0$. At the same time, the degree of purification achieved is lower than the values corresponding to the ideal separation coefficients. This fact can be explained by the influence of non-equilibrium processes, where non-volatile impurities are captured by the steam flow of the base metal, and by their concentration in the near-surface layer.

The results presented in Ref. [14] indicate the moderate efficiency of the cadmium distillation purification of impurities in the presence of Cd oxide, which can be considered as one of the stages of a complex technological scheme for obtaining high-purity cadmium.

In the paper [17], the thermodynamic equilibrium of cadmium in the condensed and vapour phase and impurities is considered: each in a binary alloy with cadmium, each of the impurities in the vapour phase above a binary alloy with cadmium. When plotting partial pressure diagrams, the following assumptions were made: the activities of cadmium and impurities in the resulting condensed phases are equal to unity; impurities in cadmium do not interact with each other and their activities in the polymetallic alloy are equal to those in binary solutions based on cadmium. Due to the fact that cadmium distillation processes are carried out at 673–873 K (400–600°C), the temperature range for the study was chosen within 400–900 K.

The metal may interact with residual gases at high temperatures during refining cadmium by vacuum distillation. The gas phase may contain significant amounts of the main components of the atmosphere (nitrogen and oxygen), as well as water vapour and carbon dioxide. Cadmium is passive towards normal nitrogen $N_2$, but reacts with active nitrogen N. Cadmium nitride $Cd_3N_2$ is unstable and decomposes by oxygen at standard temperature.

The following results emerge from the studies performed in Ref. [17] on the interaction of cadmium and its impurities with gas phase components under the distillation process conditions. Cd and impurities Tl, Zn, Pb, Cu, Ni, Ag and As, dissolved in cadmium and in the vapour phase above cadmium, under cadmium distillation conditions at

700–900 K, are not oxidized by water vapour and $CO_2$. Such an interaction is possible only at pressure greater than atmospheric.

Under distillation refining conditions, cadmium interacts with residual oxygen to form a volatile oxide; refractory impurities nickel, copper, and silver, dissolved in cadmium, do not interact with oxygen under distillation conditions. Nickel in the vapour phase reacts with residual oxygen to form a lower oxide, and copper does to do a higher oxide. Silver remains in elemental form under these conditions.

Fusible impurities of lead and thallium in cadmium interact with residual oxygen and in the vacuum form oxides including higher those. Moreover, if lead oxides are non-volatile and do not affect the quality of refined cadmium, then univalent thallium oxide has a vapour pressure comparable to that of cadmium, that is, its transition to the vapour phase and into the refined metal is possible, which can cause a deterioration in metal quality. Volatile impurities (zinc and arsenic) form oxides: in the first case, non-volatile one, that does not affect the refining process, in the second case, very volatile that, which turns into the vapour phase.

In the presented article, a thermodynamic analysis of the oxidation reactions of impurities in the cadmium melt and the surrounding atmosphere was performed, the relationship between the affinity and redox processes of impurity oxides and cadmium oxide was revealed, oxidizing refining of cadmium was applied, *etc*.

The purpose of this paper was to calculate the oxygen potentials of impurity oxidation reactions during melting of cadmium in an atmosphere of inert argon gas with oxygen present in it and during distillation in vacuum, as well as to study the complex process of cadmium purification using oxidizing refining.

## 2. THEORETICAL AND EXPERIMENTAL RESEARCH

### 2.1. Thermodynamic Analysis of Impurity Oxidation Reactions during Cadmium Melting and Distillation

The removal of impurities can be carried out by the method of oxidizing fusion of cadmium which, in turn, forms with oxygen a stable oxide CdO. The efficiency of metal impurity removal from cadmium in the form of oxides can be assessed by the strength value of the oxide compounds in certain ranges of change in temperature and ambient pressure. The strength of an oxidizing compound is characterized by the value of the oxygen potential or the absolute value of the change (decrease) in the Gibbs free energy $|\Delta G_{Me\mathrm{O}}|$.

A simple model described in Ref. [18] was used to calculate the dependences of the change in Gibbs free energy $\Delta G$ on temperature during the formation of impurity oxides in the liquid and vapour phases of

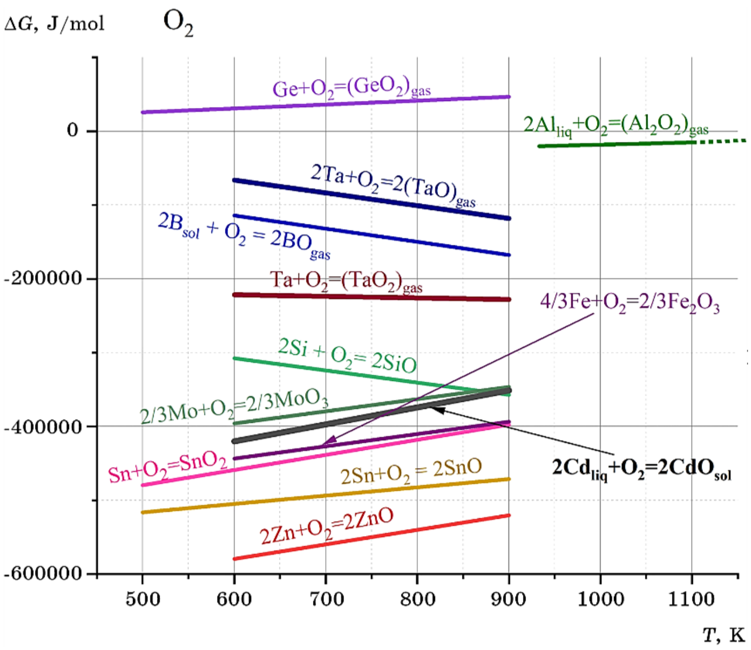

**Fig. 1.** Change in Gibbs potential at the formation of oxides of impurities Zn, Sn, Fe, Mo, Si, Ta, B, Ta, Ge in the liquid phase of cadmium during melting in an argon atmosphere.

cadmium. Figure 1 shows the temperature dependences of the oxygen potentials of the majority impurities in the liquid phase of cadmium during its melting in an argon atmosphere.

Analysis of the obtained dependences shows that impurities Zn, Sn, Fe, which the change in the Gibbs potential of oxide formation in absolute value is high, will form oxides, releasing Cd and the elements Mo, Si, Ta, B, Ta, Ge from their less stable oxides. All oxides are removed by filtering the molten cadmium, and impurities introduced into the melt during deoxidation are removed during the distillation process.

Figure 2 shows the dependences of the oxygen potentials of the majority impurities in cadmium within the studied temperature range and under the gas pressure of 0.5 Pa. The indicated curves in Fig. 2 were drawn by extrapolating the $\Delta G$ dependences obtained at the formation of oxides in the vapour phase of the high-temperature region to the studied temperature range of 600–900 K. It was assumed that all the considered impurities can transfer into the vapour phase due to the entrainment of these impurities by volatile cadmium atoms, which evaporate intensively.

In the vapour phase, the impurities such as Sn, Zn, Sb, As, and Cu form oxides that are more stable than CdO and have low vapour pres-

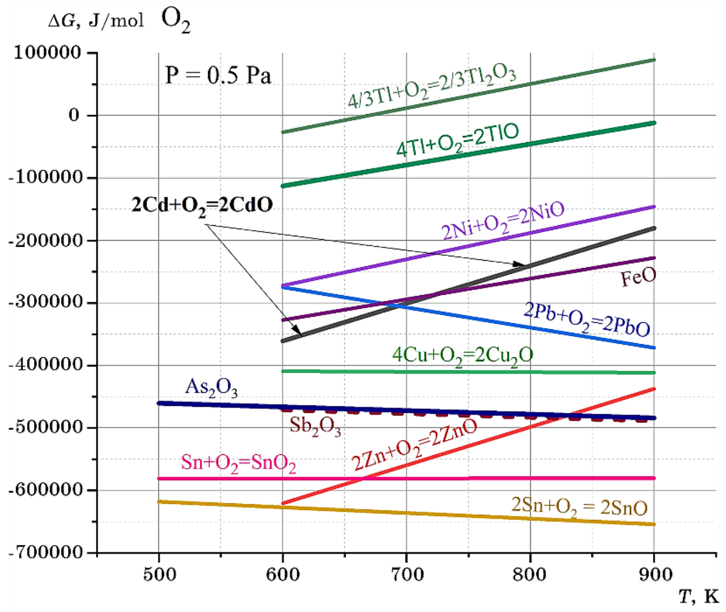

**Fig. 2.** Change in Gibbs potential during the formation of oxides of impurities Zn, Pb, Fe, Cu, Tl, Ni, As, Sn, Sb in the vapour phase of cadmium in the vacuum of 0.5 Pa.

sure. They remain as a residue in the crucible during the distillation process. Volatile arsenic oxide transfers into condensate, which can restrict the quality of the final product.

For impurities Pb, Fe, Ni, Tl, the Gibbs potential during the formation of their oxides is, in absolute value, less than the free energy of formation of cadmium oxide and metal oxides shown on the graph. The elements Pb, Fe, Ni, Tl will remain in the cadmium melt, releasing oxygen to form non-volatile oxides of cadmium and impurity attending in it. Volatile thallium oxides will enter the cadmium condensate, making fine cleaning of thallium difficult. One way to additionally remove volatile $As_2O_3$ and TlO may be using a hot condenser during the Cd distillation process, which was applied in this work.

To remove oxides formed in the cadmium condensate during its purification, the resulting distillate was melted and filtered in an environment of inert argon gas, which increases the purity of the final product.

## 2.2. Cadmium Purification Process Using a Complex Method

Analysis of literature data [13, 14, 19] shows that a simple scheme of single or multiple distillation gives a small yield (< 60%) of a suitable product, without providing high degrees of purification (> 5N). Higher purity materials are obtained by improving distillation processes, for example, the application of getter filters [4], the use of gettering impurity [14], preliminary filtration of the melt and distillation of volatile impurities [15, 20], the use of a barrier oxide layer [19], heating the surface of the condenser [21].

Crystallization refining methods (oriented crystallization, growth crystals from a melt, zone melting) are used mainly at the final stage of purification when removing small amounts of impurities from the melt. These methods can produce metals with a purity up to 99.99998 wt.% [10, 13, 21], but they can be combined with other refining methods in most cases.

In this work, the complex distillation process is investigated, including such stages as melting of the original metal and filtration in an environment of inert argon gas (a component of oxidizing refining) followed by distillation of cadmium in a vacuum from a crucible to a hot condenser (purification of non-volatile and volatile impurities).

The availability of volatile and non-volatile impurities in cadmium was determined by calculating the values of ideal impurity separation coefficients $\alpha_i$ during molecular evaporation [23] according to the formula

$$\alpha_i = \frac{p^0_{\mathrm{Cd}}\sqrt{M_B}}{p^0_B\sqrt{M_{\mathrm{Cd}}}}, \tag{1}$$

where $p^0_{\mathrm{Cd}}$, $p^0_B$—vapour pressures of cadmium and impurity $B$; $M_{\mathrm{Cd}}$, $M_B$—molecular weights of cadmium and impurity $B$, respectively.

Table 1 shows the calculated values of the ideal impurity separation coefficients $\alpha_i$ during molecular evaporation of cadmium in vacuum. The vapour pressures of the elements at specified temperatures were taken from Ref. [24]. The impurities in Table 1 above the row with Cd are classified as volatile, and below as non-volatile. Therefore, simple distillation does not provide optimal conditions for cadmium refining due to the transition of highly volatile impurities into condensate. In this regard, the processes of removing volatile impurities and purification of non-volatile impurities must be separated and carried out in a single refining cycle.

When refined cadmium is melted, surface contaminants accumulate on the surface of the melt mirror in the form of oxides, suboxides, and metal compounds, based both on Cd and impurity elements, the volatility may differ from that of pure components. The presence of surface contaminants leads to the transfer of volatile components of the oxide film into the distillate and to significant reduction in the productivity of the refining process. That is exactly why the removal of oxide film and slag from the surface of the melt during cadmium distillation is an

**TABLE 1.** Calculated values of ideal impurity separation coefficients $\alpha_i$ during molecular evaporation of cadmium in vacuum.

| Impurity | Temperature, K | | | |
|---|---|---|---|---|
| | 600 | 700 | 800 | 900 |
| Hg | $5.1\cdot10^{-5}$ | – | – | – |
| P | $7.9\cdot10^{-4}$ | $2.910^{-3}$ | – | – |
| S | $1.1\cdot10^{-4}$ | $2.2\cdot10^{-3}$ | – | – |
| Cs | $4.2\cdot10^{-3}$ | $7.6\cdot10^{-2}$ | $1.5\cdot10^{-1}$ | $2.8\cdot10^{-1}$ |
| K | $1.6\cdot10^{-2}$ | $2.1\cdot10^{-1}$ | $3.2\cdot10^{-1}$ | $3.3\cdot10^{-1}$ |
| Se | $2.5\cdot10^{-2}$ | $1.8\cdot10^{-1}$ | $1.9\cdot10^{-1}$ | $2.5\cdot10^{-1}$ |
| As | $1.8\cdot10^{-1}$ | $5.3\cdot10^{-1}$ | $2.9\cdot10^{-1}$ | – |
| **Cd** | **1** | **1** | **1** | **1** |
| Zn | 2.7 | 7.6 | 6.9 | 5.2 |
| Te | $2.2\cdot10^{1}$ | $3.2\cdot10^{1}$ | $1.8\cdot10^{1}$ | $1.4\cdot10^{1}$ |
| Na | $2.3\cdot10^{1}$ | $8.2\cdot10^{1}$ | $4.7\cdot10^{1}$ | $3.3\cdot10^{1}$ |
| Mg | $7.2\cdot10^{2}$ | $1.6\cdot10^{2}$ | $6.4\cdot10^{1}$ | $3.2\cdot10^{1}$ |
| Li | $1.3\cdot10^{3}$ | $6.5\cdot10^{3}$ | $7.3\cdot10^{2}$ | $3.2\cdot10^{2}$ |
| Sr | $5.6\cdot10^{3}$ | $7.8\cdot10^{3}$ | $2.3\cdot10^{3}$ | $9.1\cdot10^{2}$ |
| Ca | $4.0\cdot10^{4}$ | $3.9\cdot10^{4}$ | $9.3\cdot10^{3}$ | $6.1\cdot10^{3}$ |
| Sb | $4.0\cdot10^{5}$ | $2.2\cdot10^{4}$ | $3.1\cdot10^{4}$ | $7.5\cdot10^{2}$ |
| Bi | $1.0\cdot10^{5}$ | $7.9\cdot10^{5}$ | $4.7\cdot10^{3}$ | $1.7\cdot10^{1}$ |
| Ba | $1.4\cdot10^{5}$ | $1.6\cdot10^{5}$ | $3.9\cdot10^{4}$ | $1.3\cdot10^{3}$ |
| Tl | $1.3\cdot10^{5}$ | $1.4\cdot10^{5}$ | $3.0\cdot10^{4}$ | $1.0\cdot10^{4}$ |
| Pb | $7.6\cdot10^{6}$ | $4.5\cdot10^{6}$ | $7.3\cdot10^{5}$ | $1.8\cdot10^{5}$ |
| Mn | – | – | $4.3\cdot10^{9}$ | $2.2\cdot10^{8}$ |

important point in increasing the efficiency of deep cleaning.

One of the technological methods to remove oxide contaminants is filtration of liquid metal [13, 15, 18, 20]. In this regard, the task was set to study the complex process of Cd distillation, which includes the step-by-step cadmium purification of impurity oxides by filtration (component of oxidizing refining) followed by purification of non-volatile and volatile impurities by distillation of the majority element on a hot condenser.

To implement the process of stepwise purification of Cd, a distillation device is proposed, the design is schematically shown in Fig. 3.

The distillation device is made of high-purity dense graphite, which is characterized by heat resistance, minimum impurity content and chemical inertness to cadmium. The concentration of impurities in it, such as Si, Fe, Al, Mg, B, Cu, Mn, is of 1.0–0.1 ppm. The quasi-closed type distillation device consists of two identical components: the lower part serves as a crucible (*2*), and the upper that (*1*) is a collector (*D*) for

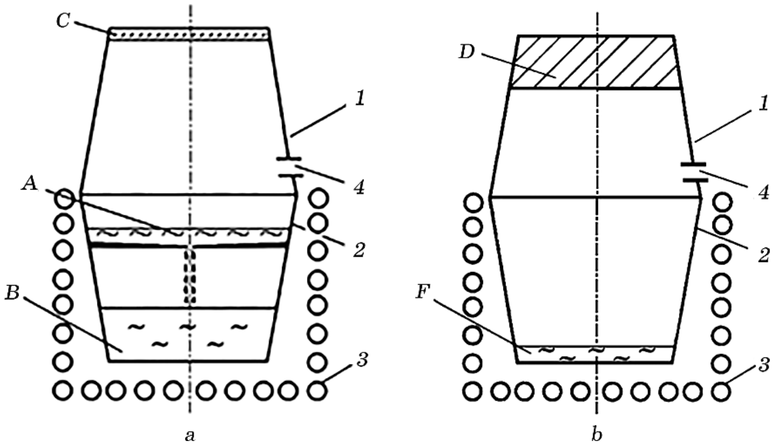

**Fig. 3.** Scheme of the device for cadmium distillation: *a*—filtration stage and partial removal of volatile impurities; *b*—stage of purification of non-volatile and volatile impurities by distillation of the metal onto a heated condenser. *1*—condenser; *2*—crucible; *3*—heater; *4*—outlet hole. *A*—source metal; *B*—filtered metal; *C*—a thin layer of condensate with volatile impurities; *D*—distillate; *F*—residue with non-volatile impurities in the crucible.

the Cd condensation. The volume of the crucible makes it possible loading from 1.8 to 2.3 kg of initial cadmium.

The purification process was carried out in two stages. At the first stage, the initial metal was melted in an environment of technical purity inert gas argon with a volume fraction of oxygen in it 0.002% under the pressure in the installation chamber of about 1 atm. The melted cadmium flowed down through a hole in the plate into the crucible (filtration process). At the same time, surface oxides and slags of the melt remained on the inclined surface of the plate, and volatile impurities (Na, K, S, P, Cl, Se, *etc.*) partially evaporated from the melt onto the inner surface of the condenser (*C*) (Fig. 3, *a*).

At the second stage, the removal of non-volatile impurities (Cu, Fe, Si, Ni, Co, V, Cr, Au, Ag, Al, Tl, Sb, Bi, Li, Sn, Mn, *etc.*) was carried out by distilling filtered metal, poured into the crucible, onto a heated condenser. During the evaporation process, the cadmium condensate is purified of non-volatile impurities that remain in the residue *F* at the bottom of the crucible (Fig. 3, *b*). In this case, volatile impurities are removed through the outlet because the condenser temperature is too high for volatile impurities to condense.

The temperature of the distillation process is higher than the melting point of Cd (594 K), and the condensation temperature is lower than that.

This combination of purification stages significantly increases the

**TABLE 2.** Content of majority impurities in initial cadmium, after filtration and vacuum distillation (distillate).

| Impurity | Initial Cd | Cd after filtration | Distilled Cd |
|---|---|---|---|
| | Impurity, $\times 10^5$ wt.% | | |
| Zn | 20 | 6 | 3 |
| Pb | 440 | 110 | 4 |
| Fe | 20 | 4 | 3 |
| Cu | 420 | 80 | 5 |
| Tl | 180 | 45 | 12 |
| Ni | 200 | 60 | 2 |
| As | 60 | 20 | 5 |
| Sn | 120 | 40 | 8 |
| Sb | 100 | 20 | 4 |
| Cd, wt.% | 99.98 | 99.992 | 99.9995 |

purification efficiency, process productivity, and the yield of a suitable product, which is at least 95% of the initial mass.

## 3. RESULTS AND DISCUSSION

Table 2 presents the results of the analysis of the impurity content in cadmium: initial, after filtration and vacuum distillation. The analysis was performed by high-resolution laser mass spectrometry. The random error of the analysis results is characterized by a relative standard deviation of 0.15–0.30.

The content of other impurities not listed in Table 2 is below the detection limit of the analysis technique—$(3–6)\cdot 10^{-6}$–$(2–4)\cdot 10^{-7}$ wt.%.

After filtration (oxidizing refining), the purity of cadmium increases by almost one order of magnitude (from the initial 99.98 wt.% to 99.992 wt.%). The efficiency of this purification method is 3–5 times for all impurities listed in Table 2.

As can be seen from the data presented in Table 2, the comprehensive method of cadmium refining is effective for the entire range of impurity elements.

The concentration of most impurities is reduced by one or two orders of magnitude. For example, the concentration of lead and nickel is reduced by 100 times and copper by 84 times.

Analysis of the results shows that the comprehensive process of cadmium purification with oxidizing refining and single distillation in vacuum with simultaneous distillation of non-volatile and volatile impurities provides almost 100-fold purification of Cd in terms of the total content of impurities.

## 4. CONCLUSIONS

The literature review carried out shows that oxidizing refining for cadmium has been investigated to some extent, but further study of this process is required.

Stable and unstable impurity oxides in relation to the main cadmium oxide were determined. It has been shown that, during the distillation process, the impurities form both non-volatile and volatile oxides; the removal requires the use of special techniques. To remove non-volatile oxides, it is proposed to filter the cadmium melt in the argon atmosphere with oxygen present in it. Moreover, to remove volatile arsenic and thallium oxides in the vapour phase, cadmium should be condensed on a hot condenser.

The results of experimental studies of the comprehensive process of distillation refining of cadmium, which includes such stages as oxidizing refining and cadmium purification of non-volatile and volatile impurities by distillation of cadmium in a vacuum from a crucible on a hot condenser, are presented. Studies have shown that the efficiency of such a process is almost two orders of magnitude (from the initial 99.98 wt.% to 99.9995 wt.%).

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