Work Safety During Purification of Irradiated Beryllium by Chlorination

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Abstract. The paper considers safety ensuring issues during the work at the plant intended for researching the technology of irradiated beryllium purification using chlorination method. Presented technology assumes handling beryllium and its compounds, chlorine and hydrogen chloride in compliance with the following safety aspects: radiation and toxic safety. Based on the results of the calculation of an expected dose and taking into account the requirements for the attended personnel, the allowable duration of maintenance the reaction chamber with the loaded irradiated beryllium sample was determined. The amount of radioactive beryllium in each cleaning cycle was determined from the point of view of the reasonable minimization (based on the safety rules) of the possible internal exposure of personnel due to the intake of gaseous tritium. The paper describes a unique way to measure the concentration of free chlorine by optical method and methods to protect the harmful effects of chlorine gas and hydrogen. The results of this study demonstrate possibility of safe technology implementation during preparation and conduction of experiments for the treatment of irradiated beryllium.

1. Introduction

Beryllium causes great interest for reactor and thermonuclear technology due to its unique neutronic features: low neutron absorption cross section and high moderating properties. Therewith, there are some problems for beryllium that restrict duration of its use as a part of nuclear facilities.

In the course of a reaction chain, gaseous nuclides such as $^4$He, $^3$He, $^3$H, $^1$H are produced on beryllium and primary reaction products, which cause the fission-gas-induced-swelling. In research reactor beryllium has to be replaced every 8-10 years, in fusion reactors it may need to be replaced every 5 years of operation [1-3].

The low beryllium content in nature shifts the focus on reprocessing of irradiated beryllium. Formation and accumulation of radioactive isotopes during beryllium irradiation is important in this process. Among them there are beryllium activation products ($^3$H, $^{10}$Be) and impurity activation products in initial beryllium ($^{137}$Cs, $^{108m}$Ag, $^{66}$Co, $^{14}$C, $^{94}$Nb).

The papers [2, 4, 5] are devoted to beryllium purification from radionuclides focusing on thermo desorption methods during tritium removal.

The article discusses safety ensuring issues during implementation of technology for beryllium purification from the cobalt, tritium, cesium and silver radionuclides, based on chemical transport reaction with gaseous chloride [6, 7].
The purpose of this work is the safety justification during experiments at the irradiated beryllium purification unit

2. Irradiated beryllium sample specification

The discussed experimental research, which are directed to create an efficient technology for irradiated beryllium purification from radioactive isotopes, are implemented using beryllium that has been irradiated in Japanese Research Reactor JMTR in 1968-1975 [8]. Thermal neutron flux density during reactor running was $8.0 \times 10^{13} \text{n cm}^{-2} \text{s}^{-1}$, fast neutron flux density was $\sim 7.5 \times 10^{12} \text{n cm}^{-2} \text{s}^{-1}$.

Table 1 presents composition of radionuclides and its activity in beryllium sample with mass of 0.96 kg at the time of delivery (July 2009) and since the moment of research start (July 2018).

Table 1. Specification of radionuclides in beryllium rod.

| Radionuclide | Nuclear reaction | Activity, Bq |
|--------------|------------------|--------------|
| $^3\text{H}$ | $^9\text{Be} (n,\alpha) ^4\text{Li}(n,\alpha)$ | $2.5 \times 10^{11}$ | $1.5 \times 10^{11}$ |
| $^{55}\text{Fe}$ | $^{54}\text{Fe} (n,\gamma)$ | $4.5 \times 10^6$ | $0.45 \times 10^6$ |
| $^{59}\text{Ni}$ | $^{58}\text{Ni} (n,\gamma)$ | $3.7 \times 10^6$ | $3.7 \times 10^6$ |
| $^{60}\text{Co}$ | $^{59}\text{Co} (n,\gamma)$ | $2.7 \times 10^8$ | $0.827 \times 10^8$ |
| $^{108m}\text{Ag}$ | $^{107m}\text{Ag} (n,\gamma)$ | $2.4 \times 10^7$ | $2.28 \times 10^7$ |
| $^{109}\text{Be}$ | $^9\text{Be} (n,\gamma)$ | $2.9 \times 10^7$ | $2.9 \times 10^7$ |
| $^{137}\text{Cs}$ | $^{235}\text{U} (n,\text{PD})$ | $1.8 \times 10^7$ | $1.46 \times 10^7$ |

We have found that radionuclides listed in the Table 1 represent major danger. For example, cobalt $^{60}\text{Co}$, which is defined by the hardest gamma-irradiation and the second largest activity; tritium $^3\text{H}$ with maximum activity; silver $^{108m}\text{Ag}$ – the second largest gamma-irradiation intensity and cesium $^{137}\text{Cs}$, which is close in activity to $^{108m}\text{Ag}$. Table 2 provides radiation characteristics of these radionuclides.

Table 2. Radiation characteristics of $^{60}\text{Co}$, $^3\text{H}$, $^{108m}\text{Ag}$ and $^{137}\text{Cs}$ [9].

| Nuclide | Type of radiation | Radiation energy, MeV | $T_{1/2}$, years | Decay, % |
|---------|-------------------|-----------------------|------------------|---------|
| $^{60}\text{Co}$ | $\gamma$ | 1.33251 | 5.27 | 99.9856 |
| | $\beta$ | 1.17323 | 99.9736 | 100 |
| | | 0.31788 | 0.4339 | 89.88 |
| $^{108m}\text{Ag}$ | $\gamma$ | 0.6144 | 438.00 | 90.39 |
| | | 0.7229 | 0.905 | 94.6 |
| $^3\text{H}$ | $\beta$ | 0.018594 | 12.28 | 89.98 |
| $^{137}\text{Cs}$ | $\gamma$ | 0.66165 | 30.17 | 94.6 |

3. Irradiated beryllium purification technology

The references [10, 11] provided the information regarding possibility to use chemical transport reactions for beryllium, titanium and uranium purification, which proposed chemical transport involving halogenide – gaseous sodium chloride (Equation 1):

$$\text{Be} + 2\text{NaCl} (g) = \text{BeCl}_2(g) + 2\text{Na}(g)$$  \hspace{1cm} (1)

Production of high purity beryllium by beryllium oxide chlorination with carbon tetrachloride was considered in 1961 [12]. It was shown that such beryllium is feasible for in nuclear engineering without any additional purification.
A case in point is a chlorination of the above mentioned beryllium sample in gaseous chlorine medium, wherein its radionuclides (gamma-emitters \(^{60}\text{Co}, \, ^{137}\text{Cs}, \, ^{108m}\text{Ag} \) and \(^{3}\text{H}\) beta-emitter) are converted into chlorides along with beryllium.

The technology of separation of beryllium and radionuclides is based on the differences in temperature limits of phase transformations of beryllium chloride and chlorides of radionuclides from solid (or liquid) to gaseous state (Table 3).

### Table 3. Chlorides melting and boiling temperatures [13-15].

| Chloride   | CoCl\(_2\) | BeCl\(_2\) | CsCl | AgCl  | HCl*  |
|------------|------------|------------|------|-------|-------|
| T\(_\text{melting}, \, ^0\text{C}\) | 724        | 404        | 646  | 455   | -114  |
| T\(_\text{boiling}, \, ^0\text{C}\)  | 1049       | 500±20     | 1302 | 1554  | -85   |

*for standard conditions (25 °C, 100 kPa).

Components of the technological process of irradiated beryllium purification from radionuclides, the main ones of which are the stages to produce beryllium chlorides, and their subsequent separation, are presented in Figure 1.

![Figure 1. The scheme of chloride production and separation (the phase state of the reagents is indicated geometrically: through a rectangle - solid, through an oval - gaseous).](image)

When interacting with chlorine in the reaction chamber at a temperature of \(~ 730^0\text{C}\), chlorides of radionuclides \(^{60}\text{Co}, \, ^{137}\text{Cs}, \, ^{108m}\text{Ag} \) and \(^{3}\text{H}\) are generated along with beryllium chloride.

In the afterburner, unreacted chlorine in the reaction chamber is bound by hydrogen to form hydrogen chloride. Hydrogen is supplied in excess to completely eliminate the presence of chlorine in the reagent mixture. Due to the exothermic reaction, temperature of the reagents in the afterburner rises to \(~ 800^0\text{C}\), and then the mixture of chlorides and excess hydrogen is cooled in the heat exchanger to \(~ 460^0\text{C}\).

Gamma-emitters (CoCl\(_2\), CsCl, AgCl) are deposited on a filter assembled from nickel rods, and beryllium chloride BeCl\(_2\) is deposited in a storage in the liquid phase as its temperature decreases to \(~ 420-480^0\text{C}\).

Gaseous hydrogen chloride and tritium chloride enter the tritium chloride storage tank, where they dissolve in water to form hydrochloric acid.

Hydrogen gas is removed from the tritium chloride storage tank through the ventilation system. To implement the described technological process, an experimental plant has been designed.

In the considered technology of processing irradiated beryllium using chlorination, the following safety aspects can be highlighted:
- radiation safety;
- toxic safety when handling beryllium and its compounds, chlorine and hydrogen chloride.
4. Radiation safety assurance

4.1. Protection of personnel from external exposure
The main method of protection from external exposure is to limit stay period of personnel in the radiation zone of the purifying plant.

Using the MCNP code [16], we calculated expected dose on the skin and the lens of a person's eye was estimated at its different positions relative to the beryllium sample in the variant of moving the person from the reaction chamber in perpendicular to the camera axis, where the beryllium sample is placed. The characteristics of the radiation of beryllium sample were taken in accordance with Table 1.

Based on the results of the calculation (Figure 2) and the requirements [17] for the A and B-group personnel, the allowable duration of reaction chamber maintenance of the plant with the irradiated beryllium sample loaded into the reaction chamber was determined (Table 4).

![Figure 2](image_url)

**Figure 2.** The dependence of the irradiation characteristics of human organs at the distance.

**Table 4.** Permissible duration of work of the personnel of A and B groups when servicing the reaction chamber of the plant depending on the distance to the beryllium image.

| Distance, cm | Personnel A, h/year | Personnel B, h/year |
|-------------|---------------------|---------------------|
| 1           | 1                   | 0                   |
| 5           | 5                   | 1                   |
| 42          | 133                 | 33                  |
| 80          | 437                 | 109                 |
| 100         | 608                 | 152                 |

If we assume that the average distance of personnel from the chlorination unit during the work is 40-50 cm, and the time required to carry out work on treating one beryllium sample is 2 hours, then, according to the data in Table 5, during a year, the A-group personnel can perform 50 treatments of irradiated beryllium samples (the average frequency of work is 5 working days), being continuously close to the plant.
4.2. Protection from internal exposure.

Provided that the experimental plant is sealed during its operation, only tritium and tritium chloride can remain in gaseous form at all stages of the implementation of the technology for beryllium purification from radionuclides.

External exposure with tritium is not dangerous, since the maximum range of tritium beta-particles in air is 5.8 mm (at 20 °C), and in biological tissue it is 6.5 μm. Therefore, beta particles of tritium are completely absorbed by the horny layers of the skin [18].

Tritium is dangerous if it enters the body through the skin, lungs or when eating and drinking water.

Tritium can release from the beryllium purification plant in case of violation of its operation modes. The most dangerous is the release of tritium with water vapor (or water particles) from tritium chloride storage tank. This process can occur due to integrity destruction of the plant, opening the tank with stored mass of water and simultaneous abnormal overflow of water.

Table 5 shows the main values of the quantities characterizing the limits of the intake of the average annual volume activity of tritium for tritiated water vapor and for gaseous tritium [17] based on the staff dose limit, which is 20 mSv.

Table 5. Values of dose coefficients, limit of annual intake with air and permissible average annual volume activity in the air of individual radionuclides for personnel.

| Radionuclide | Half-decay period | Type of compound during inhalation | Dose Coefficient, Sv/Bq | Annual intake limit for person, Bq per year | Permissible annual volume activity for person, Bq/m³ |
|--------------|-------------------|-----------------------------------|-------------------------|---------------------------------------------|--------------------------------------------------|
| H-3          | 12.3 years        | Water vapor                       | 1.8·10⁻¹¹               | 1.1·10⁰⁹                                    | 4.4·10⁶⁵                                         |
|              |                   | Gaseous tritium                   | 1.8·10⁻¹⁵               | 1.1·10¹³                                    | 4.4·10⁰⁹                                         |

In the case of full release of gaseous tritium from the beryllium sample, its activity will exceed the annual intake limit (AIL), the volume activity of tritium in the air (~ 5.5 · 10¹⁰ Bq·m⁻³ at room volume ~ 200 m³) will be significantly exceed the volume of permissible average annual volume activity (PVA).

Requirements for PVA non-exceedance is implemented if beryllium purification would be divided into several stages (not less than 13) with removal of accumulated tritium chloride at each stage. In this case, the requirements for non-excess of PVA will be met.

Then, even assuming that all tritium enters the room volume, its volumetric activity will not exceed ~ 4.3 · 10³ Bq/m³. Taking into account the value of the annual volume of air inhaled by a person equal to 2.4 · 10⁵ m³ [19], and with an experiment duration of 2 hours, the maximum intake of tritium into the lungs of one employee during one cleaning will be 2.36 · 10⁹ Bq, which exceeds the AIL.

Thus, in spite of the fact that estimates of the internal supply of tritium are conservative in nature, since a hypothetical case of the complete release of all tritium into the volume of the room in which the beryllium purification plant is located is considered, in order to ensure safety, it should be possible to exclude the presence of personnel in indoors during experiment.

5. Radiation safety during waste handling

As part of the purification plant, two main areas can be distinguished, where radioactive substances isolated from the sample of irradiated beryllium will be concentrated: chlorides in the solid phase on the surface of nickel filter rods; and tritium chloride dissolved in water in tritium chloride storage.

Chlorides of ⁶⁰Co, ¹⁰⁸Ag, ¹³⁷Cs and chlorides of less active radionuclides can be washed off nickel rods using their solvents (CoCl₂, CsCl – water, methyl and ethyl alcohols; AgCl – aqueous solution of ammonia, cyanides, rodanides, alkali metal thiosulfates; pyridine and liquid ammonia). The resulting liquid radioactive solutions can be reprocessed for immobilization and preparation for long-term storage and subsequent disposal (evaporation, cementation, etc.).
The gamma-emitter concentration in the waste from the surface of the filter will be higher than initial concentration in the beryllium rod. When handling these wastes, it is desirable to divide them into several parts to reduce the risk of personnel exposure.

When working with a solution containing tritium, the main danger lies in tritium release from the volume of the tank with the solution. The lowest maximum energy of beta particles 16 keV and the average energy 5.7 keV minimize the risk of handling the solution in the storage tank.

6. Toxic safety assurance

6.1. Safety assurance when handling beryllium and its compounds

When handling beryllium and its compounds, it is required to take into account their toxic properties [20].

The toxic properties of beryllium are manifested in relation to all human organs, but the most dangerous is the effect of beryllium on the lungs [21, 22]. In terms of irradiated beryllium purification method under discussion, the most unfavourable from a hygienic point of view are processes with beryllium compounds vapor release and possible entry of beryllium vapors into the respiratory system of personnel, as well as the stages of method implementation, where beryllium chloride can potentially affect the skin of personnel.

The maximum permissible concentration of beryllium in the air of industrial premises is 1.0 μg·m⁻³[23], which is too low. In this regard, the work related with release of beryllium compound vapors should be carried out in specially equipped isolated rooms with combined extract-and-input ventilation so that the amount of extracted air exceeds the amount of input air by 15-20%. The work at the site for irradiated beryllium purification must be arranged in compliance with the requirements of the rules [23], including ventilation requirements of the plant and the process room, its regular wet cleaning, and obligatory use of individual protective equipment by the personnel.

6.2. Safety assurance when chlorine handling

Chlorine is a poisonous and dangerous gas. To ensure the safety of personnel, it is necessary to exclude the possibility of chlorine releasing outside hermetic volumes of the plant. The feeding of chlorine to the reaction chamber begins after preheating the beryllium with a heater mounted on the outer surface of the reaction chamber casing. The reaction rate varies with beryllium temperature.

The reaction is carried out with an excess of chlorine. Excess chlorine is neutralized by reacting with the hydrogen fed after the reaction chamber. The hydrogen feeding to the afterburner is carried out in an amount exceeding the stoichiometric one.

Figure 3 presents a device for burning off chlorine residues. At the inlet of the cylinder-afterburner, there are holes for hydrogen feeding to the beryllium chloride stream and chlorine residues. Hydrogen with a temperature of ~ 20 °C is fed through openings perpendicular to the flow of the gas mixture from the chlorinator.

Interaction between hydrogen and chlorine in general occurs according to the Equation 2:

\[ \text{H}_2 + \text{Cl}_2 = 2\text{HCl} + 184 \text{kJ} \]  

Reaction occurs in two phases as shown by Equations 3 and 4:

\[ \text{Cl} + \text{H}_2 + 1 \text{kJ} = \text{HCl} + \text{H} \]  

\[ \text{H} + \text{Cl}_2 = \text{HCl} + \text{Cl} + 188 \text{kJ} \]
Figure 3. Chlorine residue burnout scheme.
1 – reagent dispenser; 2 – reactor chamber (chlorinator) casing; 3 – heater; 4 – holes for hydrogen feeding into mixer; 5 – mixer casing (afterburner); 6 – filter; 7 – interconnection joint of mixer and filter; 8 – flange connector.

Photochemical dissociation of chlorine molecules into atoms is caused by light with a wavelength of 550 nm. The activation energy of the first of these reactions is 25 kJ·mol$^{-1}$, and the second one is 8 kJ·mol$^{-1}$. Small values of these energies cause rapid development of the reaction as a whole.

In a mixture of chlorine and hydrogen, these reactions can take place too quickly, up to the explosion. When inert gases (e.g., nitrogen) are added to the mixture, the reaction rate slows down. One of the inert gas as in example in this technology is beryllium chloride. When the content of the active components comprises several percent, the explosive reaction is excluded.

The chlorine content at the chlorinator output depends on many parameters of its operation. It is essential that it does not exceed 10 mass percent, and 3-5% is optimal. In the latter cases, the heat of reaction (1) will be relatively small, and reliable control of the chlorine content in the beryllium chloride stream is ensured. The content of the components for these conditions in the considered direct-flow chlorinator is presented in Table 6.

Table 6. Content of BeCl$_2$, Cl$_2$, and H$_2$ at afterburner inlet.

| Case | Chlorine in BeCl$_2$, % | Flow rate, g·s$^{-1}$ |
|------|-------------------------|-----------------------|
|      |                         | BeCl$_2$  | Cl$_2$  | H$_2$  |
| 1    | 3                       | 6.984    | 0.21    | 0.006  |
| 2    | 5                       | 6.839    | 0.36    | 0.010  |
| 3    | 10                      | 6.479    | 0.71    | 0.020  |

It follows from the above that, for safety purposes, the availability of an efficient method for measuring the amount of excess chlorine remaining after interaction with beryllium is of fundamental importance.

The method for measuring chlorine content is based on the ability of chlorine to reflect light waves with a wavelength of 589.3 nm, due to which chlorine has a yellow-green colour, while the main component of the gas flow, which is beryllium chloride, is a transparent gas. A scheme for measuring chlorine concentration, based on its optical characteristics, is shown in Figure 4.
Figure 4. Scheme for chlorine concentration measurement in the operating flow.
1 – photomatrix (or lux meter), 2 – light flux reflected from the gas mixture, 3 – lens, 4 – transparent casing of reaction chamber, 5 – gas mixture, 6 – light-emitting diodes, 7 – shield screen.

Preliminary experiments confirmed the possibility to implement the proposed measurement scheme and the satisfactory accuracy of the results obtained in the operating range of the expected excess chlorine content.

The chlorine combustion process should be carried out with an excess of hydrogen. Otherwise, it becomes possible for chlorine to enter the tritium chloride storage tank. If a gas mixture with chlorine passes through the water of the tritium chloride storage tank, chlorine will actively interact with water to form hydrochloric acid and HClO₄ (chlorine solubility in water reaches ~ 4.6 g per 1 liter of water at a water temperature of ~ 45 °C). Emission of chlorine into the atmosphere is excluded.

7. Conclusion
The following was considered as main risk factors:
- Radiation safety.
  The activities of radionuclides in irradiated beryllium were calculated during the work. The ⁶⁰Co gamma-emitter and ³H beta-emitter pose the main danger from the used irradiated beryllium in 2018. With the extension of the work, ¹⁰⁸ᵐAg gamma-emitter will pose a significant hazard.
  The characteristics of the radiation situation were determined when working with the irradiated beryllium, taking into account the actual set of radionuclides (dose rates, permissible operating time)
  The safe work of the A-group personnel is ensured with an annual processing of 50 rods of irradiated beryllium with a frequency of 1 week.
- Danger of chlorine emitting into the atmosphere.
  The elimination of chlorine emitting into the atmosphere is achieved by burning off the chlorine residues at the exit of the chlorinator site with hydrogen. Regulation of the burning process is carried out in accordance with the chlorine content at the output of the reaction chamber, measured by the reflected light flux in the spectral region with a wavelength of 589.3 nm.
  Measurement of chlorine content by this method showed high accuracy.
  Possible residues of chlorine after burning out are dissolved in the water of the tritium storage tank.
- Risk of violation of thermal and hydraulic regimes:
  Cooling of the chamber of chlorine afterburner and sealing of the chlorination reaction chamber is provided with a stream of nitrogen.
  The range of working content of chlorine residues at the reaction chamber outlet and the conditions for controlling the process of chlorine afterburning with hydrogen are determined.
It is proposed to dilute possible hydrogen residues after reburning with inert argon (or nitrogen) at the gas flow outlet from the tritium chloride storage tank.

Thus, it can be concluded that the work carried out at the irradiated beryllium purification installation is carried out taking into account the impact of harmful factors and compliance with all necessary measures, which ensures that there will be no harmful impact on the attendant personnel.

Acknowledgment
This work was carried out with the financial support of the Committee of Science of the Ministry of Education and Science of the Republic of Kazakhstan State Institution under the Agreement 305 dated 03.30.2018 on the theme “Improving the method of irradiated beryllium dry purification”.

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