

Production of a highly enriched ^{176}Yb isotope in weight amounts by the atomic-vapour laser isotope separation method

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Abstract. The production of a highly enriched ^{176}Yb isotope in weight amounts by the atomic-vapour laser isotope separation method is reported. The content of ^{176}Yb achieved in the experimental samples of the enriched material was 99 %. For a commercial production of about 20 mg h^{-1} , the concentration of this isotope is about 88 % with about 5 % of the 'harmful' isotope ^{174}Yb . The experimental results may serve as the basis for the technology of obtaining ^{177}Lu isotope for applications in modern nuclear medicine as a drug with a high specific activity.

Keywords: laser isotope separation, ytterbium, high enrichment.

1. Introduction

Atomic-vapour laser isotope separation (AVLIS) has been developed intensely during the last decades (see, for example, reviews [1–5]) and offers considerable promise for the rare isotopes of elements that do not have stable gas-phase chemical compounds, which rules out their enrichment by gas centrifugation [6]. At present, considerable progress has been made in obtaining the rare ^{168}Yb isotope (see [7–9] and reviews [1, 3, 5]).

At the same time, it is important to enrich not only the rare ^{168}Yb isotope (having a natural abundance of 0.14 %), but also the comparatively abundant ^{176}Yb isotope (12.7 %). This isotope is used for obtaining the radionuclide ^{177}Lu (with a half-life of 6.734 days) which is quite prospective for use in modern nuclear medicine as a drug with a high specific activity. This isotope can be obtained, for example, by bombarding the initial material containing ^{176}Yb by neutrons from a nuclear reactor.

The requirements imposed on the isotopic purity of the initial material, which ensure the production of ^{177}Lu , were formulated in Ref. [10] and mainly boil down to limiting the concentration of ^{174}Yb (31.8 % in the natural isotopic mixture) strictly to the level 2 %–5 %. Accordingly, the

concentration of the target isotope ^{176}Yb in the material, which is responsible for a high specific activity of the radionuclide ^{177}Lu , must be high. Thus, the problem is reduced to obtaining a material comprised practically of the monoisotope ^{176}Yb , i.e., a highly enriched isotope whose concentration in the natural mixture is close to 100 %. Such a high enrichment can usually be attained with the help of electromagnetic separators, and the cost of isotopic materials obtained by this method is extremely high [11].

The AVLIS method make high enrichment possible in principle [1–5]. However, cases of their practical application for commercial production of highly enriched isotopes with the required concentration of the target isotope were not known until recently. The first report on the recovery of highly enriched ^{176}Yb isotope (with a concentration of 95 %) by the method of laser isotope separation appeared only recently [12].

In this paper work, we report the production of the ^{176}Yb isotope with a concentration of up to 99 %. For a device with a productivity of about 20 mg h^{-1} , the concentration of this isotope is about 88 % with about 5 % of the ^{174}Yb isotope.

2. Preliminary remarks

Isotopic and hyperfine structure of Yb transitions was studied experimentally in the beginning of 1990s [13]. As a whole, the data presented in these papers corresponds to the results obtained in the development of technology for production of highly enriched rare isotope ^{168}Yb in weight amounts [1, 3, 5, 7–9].

Preliminary analysis of the structure of transition lines upon three-stage selective photoionisation of Yb showed that the high selectivity of photoexcitation of the ^{176}Yb atoms for a residual Doppler linewidth of about 1 GHz is possible only when two narrow-band lasers (and not one, as reported in [1, 3, 5, 7–9]) with a spectral width not exceeding 500 MHz are used. At the first stage of the transition, the excitation of all isotopes except ^{176}Yb and ^{174}Yb is suppressed. At the second stage, the excitation of the isotope ^{174}Yb is suppressed.

The exact values of laser frequency detuning at both stages was determined experimentally by using a magneto-static mass spectrometer. The laser radiation wavelengths at the first and second stages of excitation were selected in such a way that the ^{174}Yb and ^{173}Yb isotope impurity concentration was minimal (not exceeding 5 %). The laser radiation intensity at the mass spectrometer input corresponds to the values used in commercial production. This

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condition is important since the selectivity of photoionisation depends on the intensity of laser radiation. An excessive increase in intensity at selective transitions leads to a field broadening of lines and excitation of undesired isotopes [9].

Mass spectroscopic measurements reveal that the total impurity concentration can be maintained stably at the level 3%–4% for radiation power density values 1–3 W cm^{-2} (first excitation stage), 2–5 W cm^{-2} (second excitation stage), and 8–25 W cm^{-2} (third excitation stage). Figure 1 shows the characteristic mass spectrum of Yb obtained under these conditions of irradiation. A lowering of the intensity of laser radiation by a factor of 8–10 led to an even higher selectivity of photoionisation with a ^{176}Yb concentration of about 99%. In this case, the photoionisation of atoms decreased considerably. Such a regime, accompanied by a decrease in productivity, was apparently realised in [12] where the possibility of obtaining highly enriched ^{176}Yb isotope was demonstrated.

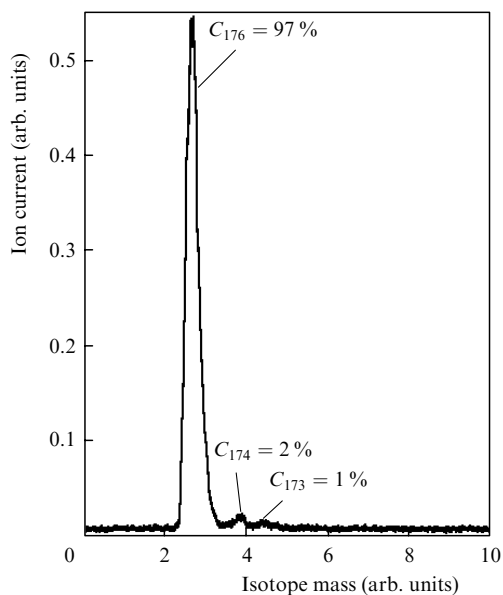


Figure 1. Mass spectrum of ytterbium for optimal wavelengths in both narrow-band channels.

Even the highest selectivity of photoionisation attainable in a mass spectrometer may turn out to be inadequate for obtaining highly enriched materials with the required concentration of isotopes, since extractor contamination processes play a significant role in experiments aimed at commercial production of enriched isotopes [1, 3, 5, 14–17].

Contamination may be caused by ions of isotopes whose spectrum is closest to that of the target isotope due to Doppler broadening of atomic lines. Collimation of the atomic beams may be used for reducing the Doppler linewidth. In this case, the linewidth decreases in proportion to $\sin \beta/2$ (β is the angular divergence of the atomic beam) (see [5, 17] for details). In the separation module (Fig. 2), the beam collimation in the section along the laser beam is small ($\beta = 120^\circ$), and hence the residual Doppler linewidth is ~ 950 MHz (i.e., an order of magnitude higher than in the mass spectrometer). However, an analysis of the structure of selective transition lines shows that the effect of Doppler broadening can be disregarded for optimal wavelengths.

If direct entry of atoms from the atomic beam into the extractor is precluded, the extractor is contaminated as a result of resonance charge transfer of ions at the vapour atoms, scattering of atoms during collisions in the beam, as well as scattering of atoms at the structural elements.

The cross section σ for resonance charge transfer of Yb at thermal velocities amounts to $\sim 2 \times 10^{-14}$ cm^2 [18]. Consequently, the charge transfer probability for a characteristic distance $d \approx 5$ cm from the extractor and a vapour density $N \times 10^{12}$ cm^{-3} is $p = 1 - \exp(-\sigma d N) \approx 10\%$. A value of $p < 5\%$ can be attained by reducing the vapour density N to a value smaller than 5×10^{11} cm^{-3} .

Contamination of the extractor due to infrequent overtaking collisions was found to be significant during separation of rare isotopes having a very low concentration relative to other isotopes [1, 3, 5, 14–17]. According to [14], the mass flux of neutral atoms in an extractor with an input slit area of 100 cm^2 is ~ 0.1 mg h^{-1} for an average number density 10^{12} cm^{-3} of atoms in the beam in the given experimental geometry. Such a flux corresponds to an ‘equivalent current’ of 0.02 mA. Consequently, the fraction of contaminating isotopes does not exceed 1% for an ion current of more than 2 mA in the extractor. Contamination associated with scattering from the structural elements can be reduced considerably by using an appropriate design of the extractor and the trap for the vapour flow [1, 3, 5].

The above contamination processes are just like a background in experiments on commercial production of the isotope ^{176}Yb . In other words, the concentration of undesired isotopes is proportional to their natural abundance. Assuming that in this case the film at the collector surface is formed by the flow of ions and atoms whose composition is in accord with the natural abundance of the isotopes, we can restore the real isotopic composition without the background contamination characterising the actual selectivity of photoionisation during the production of the target isotope. The corresponding concentration C_{real} of the isotopes can be defined by the expression

$$C_{\text{real}} = (1 + \alpha)C_m - \alpha C_{\text{nat}},$$

where C_m is the measured concentration of the isotope; C_{nat} is the natural isotope concentration; and α is the contamination factor (in units of atoms ion $^{-1}$). The parameter α is determined by considering that $C_{\text{real},i} = 0$ for the i th isotope whose atomic number differs considerably from that of the target isotope. Thus, laser radiation does not ionise the i th isotope which is introduced only by the flow of neutral atoms of natural composition. In this case, we have

$$\alpha = \frac{C_{m,i}}{C_{m,i} - C_{\text{nat},i}}.$$

Practical application of such a procedure for analysing the mass spectra leads to satisfactory results for a high accuracy of measurement of isotopic composition and moderate ($\alpha < 10$ atoms ion $^{-1}$) contaminations. In this case, the value of C_{real} is usually stable and independent of i for several isotopes whose atomic numbers differ considerably from that of the target isotope. The procedure can be used for obtaining a quantitative estimate of the contamination level from the results of isotope analysis at various spots in the extractor and in the washed enriched material. The impurity concentration in the sample is

determined in this case by the parameter $C_{\text{imp}} = \alpha/(1 + \alpha)$. A contamination factor $\alpha \leq 0.05 - 0.1$ atoms ion^{-1} must be ensured for solving this problem. Such stringent requirements can be met by using a moderate vapour density and a high efficiency of ion collection by the extractor, so that the ion current exceeds the 'equivalent contamination current' by more than an order of magnitude.

3. Experimental setup

Figure 2 shows the experimental setup for producing the ^{176}Yb isotope. It consists of the following components: a copper vapour laser (CVL) system, a tunable dye laser (DL)

system, a mass spectrometer, a separation module, and a system for controlling, monitoring and data processing (CMDP).

The three-channel CVL system was used for pumping the cells of the DL system. Kristal LT-40 sealed tubes with antireflection coating on the windows were used as the active element of the master oscillator (MO) and amplifiers. Pumping was performed by the 510-nm line filtered by mirrors of the system and filters (not shown in Fig. 2). Telescopes were used for matching the aperture of beam diameters with the cell size.

Through-pass power meters (TPMs) coupled with the CMDP system through fibreoptic transmission lines were

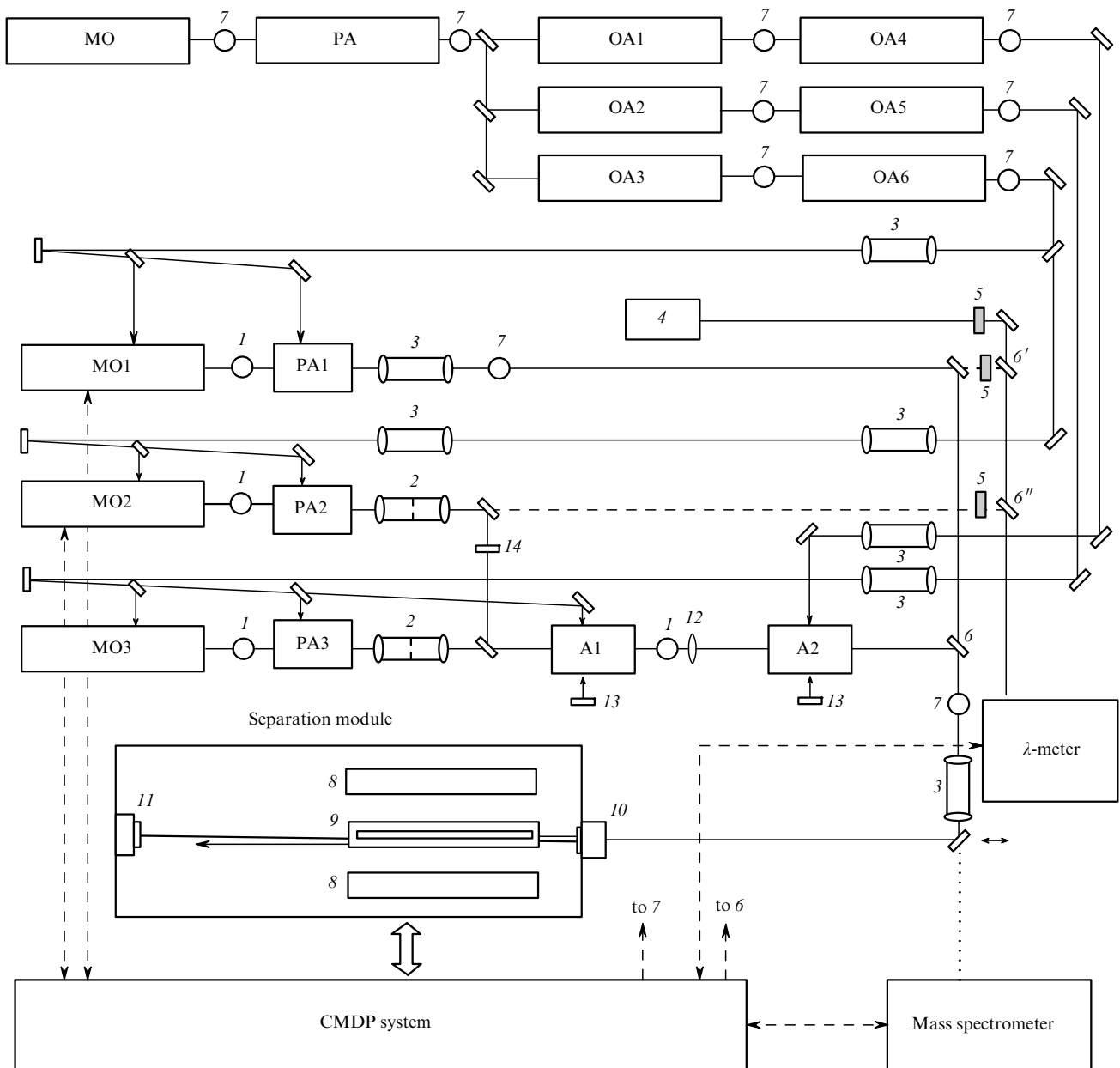


Figure 2. Scheme of the experimental setup for laser separation of ytterbium isotopes: MO – master oscillator of the CVL system; PA – pre-amplifier of the CVL system; OA1–OA6 – output amplifiers of the CVL system; MO1 – MO3 master oscillators of the dye laser (DL) system; PA1 – PA3 – pre-amplifiers of the DL system; A1, A2 – amplifiers of the DL system; (1) polariser; (2) spatial filter; (3) lens telescope; (4) stabilised He–Ne laser; (5) electromagnetic switch; (6, 6', 6'') dichroic mirrors; (7) through-pass power meter; (8) extractor; (9) vaporiser; (10) unit for composite beam introduction; (11) reflector unit; (12) lens; (13) cylindrical mirror; (14) step attenuator.

used for synchronisation of the amplifiers and for monitoring the power and pulse shape. The output power in the CVL system channels in the experiments was 20, 30, and ~ 45 W. The pulse FWHM was ~ 20 ns for a laser pulse repetition rate of 11 kHz.

The DL system with the transverse pump geometry consisted of three frequency-independent channels (corresponding to the three-stage mode of photoionisation of Yb), each containing an MO (MO1–MO3) and a preamplifier (PA1–PA3). The laser beams from the second and third channels (with comparable wavelengths) were spatially integrated at the PA2 and PA3 outputs with the help of mirrors and telescopic spatial filters for subsequent synchronous amplification of laser pulses in the amplifier stages A1 and A2. A two-way pumping of the cells was carried out in amplifiers with the help of cylindrical mirrors (13).

The radiation power at the amplifier output was controlled by TPMs and the corresponding CVL system meters. The ratio of the radiation powers from the second and third channels was monitored at the output of the combined channel by step attenuator (14). Telescopes and dichroic mirror (6) were used to combine the output laser beam with the radiation beam from the first channel to form the composite beam being introduced into the separation chamber.

The wavelength and the emission spectrum (500 MHz in narrowband channels) of each stage were formed by independent master oscillators whose design envisages the possibility of smooth tuning and automatic maintenance of frequency with an accuracy not worse than ± 50 MHz per channel [9]. In the frequency maintenance mode, the step-by-step frequency stabilisation in the laser channels was carried out using electromagnetic switches (5) admitting radiation from the appropriate channel to the λ -meter. Frequency stabilisation in the third channel was not required since the transition line in the autoionisation state of Yb was much broader than the spectral linewidth of laser radiation as well as the possible shift associated with the frequency drift. Absolute frequency calibration of the wavelengths to within ± 20 MHz was ensured by a stabilised He–Ne laser. The average values of laser radiation power in the experiments did not exceed 1 W (first stage of excitation), 2 W (second stage), and 8 W (third stage) for laser pulse durations of 10, 10 and 18 ns, respectively.

A vacuum chamber containing two electrostatic extractors and an evaporator mounted on a charging car was the main element of the separation module. The vacuum chamber was equipped with units (10) and (11) for obtaining a large number of passes by laser pulses from the composite beam through the atomic beam formed by the vaporiser. A residual pressure of 10^{-5} Torr was maintained in the experiments. The vapour beam was formed with the help of a resistive graphite vaporiser and a diaphragm with a 10-mm-wide slit of length 90 cm.

The mass flow rate of vapour at the diaphragm output was maintained at the level 12–18 g h^{-1} in the experiments. The average vapour density in the photoionisation region did not exceed 10^{12} cm^{-3} .

Collection of ions was carried out by two-electrode (collector–grid) electrostatic extractors which were used earlier by us for producing ^{168}Yb isotope [1, 3, 5]. Two extractors with a grid voltage of 2.5 kV and a collector voltage up to 18 kV were used in the experiments. The isotope composition in the extractor was monitored by

molybdenum foil ‘witnesses’, while quartz sensors of the Inficon-type film-thickness meter were used for point-by-point monitoring of contamination by neutral atoms in the extractor and at its output slit. The extractor ion current in the collector circuit was measured by a milliamperimeter and a shunt connected to the input of a Tektronix TDS3034 digital oscilloscope.

The selectivity of photoionisation during execution of the technological process and in the course of the experiment was monitored with the help of a magnetostatic mass spectrometer with an inbuilt vaporiser forming an atomic beam with a residual Doppler linewidth not exceeding 100 MHz. The online control of the isotopic composition after the experiment was carried out using a dynamic mass spectrometer by vaporising a piece of the ‘witness’ cut out from the foil. Specialists from the Nuclear Reactor Research Institute carried out washing and chemical purification of the separated material, as well as a precision isotope analysis.

The system for monitoring, control and data processing (CMDP) installed in the local network of three computers was used to monitor the laser radiation power, vapour density, ion current and other main parameters of the experimental setup, as well as to measure and stabilise the wavelengths.

4. Experimental results

Trial production of ^{176}Yb isotope was carried out in a series of four experiments lasting five hours each. The main differences in the experiments were associated with slight modifications in the design of the extractors aimed at improving the electrical strength and at attaining the highest possible stable current, variation of the position of the extractors and density of the atomic beam in the photoionisation region, as well as the introduction of additional diaphragms in the path of the atomic beam.

The laser radiation intensity was varied slightly (within 20 %) in each series of experiments. The extractor (collector) current in the experiments varied from 2 to 14 mA for a grid voltage -2.5 to -3 kV and a collector voltage of -8 to -14 kV. Oscillographic measurements of the ion current pulses show that the extractor collects only a small part of the plasma ions (about 10 % per extractor). Note that the efficiency of ion collection in low-density plasma attained values of 50 % [1, 3, 5, 9]. A deterioration of the electric strength of the extractors accompanied by breakdowns was observed in the course of isotope separation.

The enriched material was sprayed in the extractors from high-voltage copper collector surfaces and collected on a copper foil (ion collector) in the form of a durable film whose thickness increased with the collection time. Copper sputtered by ions from the collector surface was also deposited on the same film. The enriched material film was deposited on the foil in the form of two strips whose size and position were determined by the electric field configuration within the extractor.

After collection of the material, the foil was removed, weighed, cut into several parts, and subjected to washing, cleaning and precise isotope analysis. Weighing of the foil made it possible to determine the excess mass of the deposited material (copper and ytterbium) and to evaluate the exact mass balance using the data on washed-out ytterbium. The ‘witnesses’ removed from the foil revealed

the real isotopic composition in different parts of the extractor foil. The precise isotope analysis of the wash-out from the cut portions of the foil refined these data and provided information about the mass-number distribution of the isotope material.

The first experiments revealed the presence of a narrow zone on top of the extractor (covering 10 % of the foil area) with a higher concentration of impurity isotopes. The presence of this zone is associated with the scattering of atoms flying from the beam at large angles to the extractor surface at the input screens of the extractor. The isotopic composition of the rest of the foil was practically constant (with a ^{176}Yb concentration of about 80 %). The design of the extractor was modified in subsequent experiments in order to obtain a uniform distribution of isotopes over the entire foil surface. The main experimental results obtained during the isotope production can be summed up as follows.

1. The isotopic composition of the washed-out material was almost independent of the extractor current (in the range 2–10 mA).

2. The distribution of the isotopic composition of the washed-out material was almost independent of the coordinate along the foil and directed at right angles to the generator of the cylindrical casing of the extractor, while the mass distribution of washed-out ytterbium along the coordinate was quite nonuniform (Fig. 3).

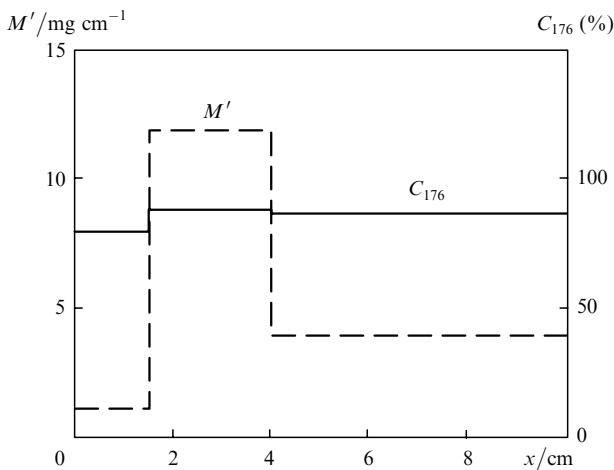


Figure 3. Dependences of the enriched ytterbium mass distribution M' and concentration C_{176} of the ^{176}Yb isotope in an extractor on the x coordinate in a direction perpendicular to the generatrix of the cylindrical casing of the extractor.

3. For identical values of the extractor current, the concentration of the target isotope increased and the concentration of impurity isotopes decreased upon a decrease in the density of atomic vapour.

4. The ratio of the 'current' mass (determined by ion current and the isotope production time) and the accumulated mass of ytterbium (obtained as a result of wash-out) varied in the range 3–5.

An analysis of the experimental data indicates that resonance charge transfer plays a decisive role in the extractor contamination. Scattering of atoms introduces only a slight variation (of just a few percent) in the isotopic composition in the regions where the ytterbium-enriched film on the foil is quite thin.

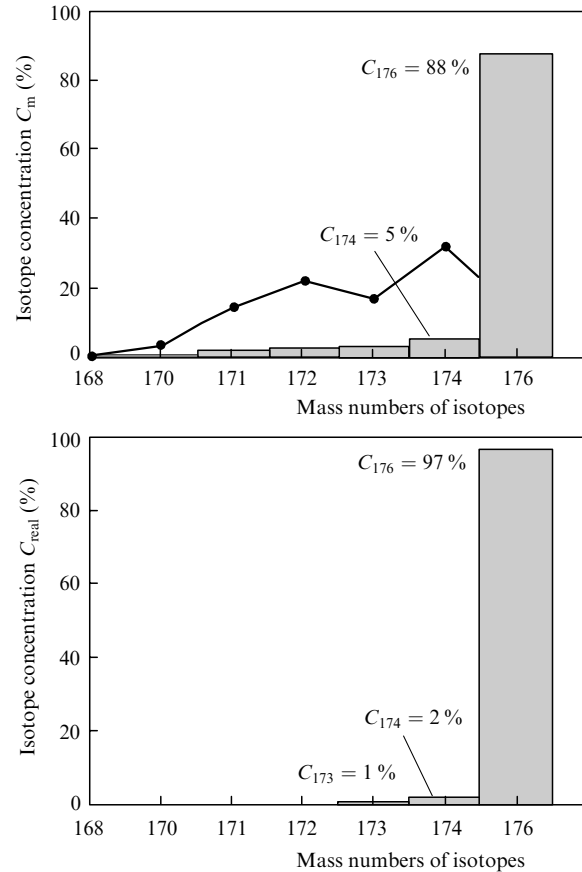


Figure 4. (a) Characteristic data (C_m) of isotopic analysis of an enriched ytterbium sample obtained from one of the extractors, and (b) the corresponding isotope composition (C_{real}) free from contamination by neutral atoms. The dark circles correspond to the natural abundance of the ytterbium isotopes.

Figure 4a shows the precise isotope analysis data for a sample of the initial material with an enriched ytterbium mass of 54.2 mg.

In this sample, the concentration of the ^{176}Yb isotope is 87.9 %, and that of the ^{174}Yb isotope is 5.3 %. The contamination factor is equal to 0.1, and the impurity isotopes have a total mass of 4.9 mg.

The sputtering coefficient K_p for copper, which is known to depend on the energy of an ion and the angle of its incidence [19, 20], was determined from the data on isotope analysis. The experimentally measured value of the sputtering coefficient varies in the range 12–14 atoms ion⁻¹ at an energy of 12 keV and almost coincides with the values obtained for sputtering of lead ions in the incidence angle interval 0–60°.

5. Conclusions

Our experimental investigations have shown that AVLIS can be used for obtaining the initial material for production of ^{177}Lu . Test samples have been obtained with up to 88 % concentration of the ^{176}Yb isotope and about 5 % of the ^{174}Yb isotope for a production rate of $\sim 20 \text{ mg h}^{-1}$.

It has been shown experimentally that resonance charge transfer of ions in an atomic beam is dominant among all processes of extractor contamination. Our estimates of its effect indicate that a decrease in the ^{174}Yb impurity

concentration to 2.5 %–3 % can be attained by lowering the vapour density to $\sim 10^{11} \text{ cm}^{-3}$ using the three-stage photo-ionisation mode or by decreasing the concentration of ^{174}Yb through an increase in the concentration of ^{173}Yb (an isotope that does not hamper the recovery of ^{177}Lu isotope).

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