Radioactive $^7\text{Be}$ targets for measurements of the cross section of the $^7\text{Be}(p,\gamma)^8\text{B}$ reaction

M. Hussonois, L. Brillard, C. Le Naour
IPN, IN2P3-CNRS et Université Paris-Sud, 91406 Orsay, France

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1 Introduction

The interpretation of the most recent solar neutrinos experiments requires a good knowledge of the cross section of the reaction $^7\text{Be}(p,\gamma)^8\text{B}$ at very small energy ($E_{\text{cm}}=18$ keV). We have recently measured\cite{1} this cross section for $E_{\text{cm}}=0.35$-1.4 MeV and for $E_{\text{cm}}=0.112$-0.190 MeV. We report here on the description of the preparation of the radioactive targets of $^7\text{Be}$ used in these experiments.

2 $^7\text{Be}$ build up

$^7\text{Be}$ was produced via the $^7\text{Li}(p,n)^7\text{Be}$ reaction at the 4 MV Van de Graaff accelerator in Bordeaux. The enriched (99%) metallic $^7\text{Li}$ target was 7 mg/cm$^2$ thick and was deposited on a water-cooled backing. The irradiation was carried on for about 30 days, with an average beam intensity of about 30$\mu$A at a proton energy of 3.2 MeV. At the end of the irradiation, the $^7\text{Li}$ target was kept under vacuum until chemical processing begins.

3 Chemical procedure.

3.1 Materials.

The contamination by macro-amounts of alkali earth elements (such as Ca, Ba), homologue of Be and difficult to separate, was avoided by use of vessels in Teflon$^R$ or in quartz, perfectly cleaned by boiling with ultra-pure nitric acid and water.
All the concentrated acids and water, already of high purity, were distilled, just before use, in Teflon\textsuperscript{R} apparatus. Diluted acids were prepared in quartz vials. The three used columns were made with Teflon\textsuperscript{R} tubes (2.4 mm inner diameter) and Teflon\textsuperscript{R} connectors. The different acids were flowed through these columns with a peristaltic pump by means of tubings in Teflon\textsuperscript{R} and/or Tygon.

The first column was filled with the di-2-ethyl hexyl phosphoric acid (HDEliP) sorbed on Teflon\textsuperscript{R} powder (Voltalef, particle diameter less than 50\textmu m). After washing with boiled distilled water (to eliminate air bubbles and excess of HDEHP), this column was cleaned from mono, di and trivalent elements by washing with 10 ml of 6M HN\textsubscript{0}\textsubscript{3}. The minimum of water was used to wash till neutrality, and finally the column was conditioned with 2 ml of 5. 10\textsuperscript{-3} HN\textsubscript{0}\textsubscript{3}.

The second column was filled with the cation exchange resin AG 50 X 8, 200 - 400 mesh previously treated to eliminate Fe impurities. Nevertheless, the column was successively washed with 6M HN\textsubscript{0}\textsubscript{3}, water and 0.1 M HN\textsubscript{0}\textsubscript{3}. The third one is a small column filled with about 50 mg of macro-porous anion exchange resin, AGMP1, which gives very few products of degradation with concentrated acid. This column was previously washed with water and 8M HCl.

The different steps of the chemical separation were monitored by measurements of all fractions by detecting the 478 keV \gamma-ray of \textsuperscript{7}Be by \gamma-spectroscopy chain.

4 Target dissolution.

The target, lithium deposit on its copper backing, conserved under vacuum till the beginning of the chemistry, is rapidly placed in the dissolution cell. This system, practically closed, was developed to avoid contamination. It is composed, at the bottom, of a Teflon\textsuperscript{R} ring, of inner diameter adjusted to the size of the lithium deposit, which prevent leakage and contact with copper and glass of the alcohol-solvent. The upper part is a glass tube, closed up by a Teflon\textsuperscript{R} flask with a small hole (diameter= 3mm) to introduce and pick up the solvent. This ensemble is pressed between two copper flasks.

The dissolution starts immediately with the introduction of a small volume (1 ml) of pure methanol. The thickness of the Teflon\textsuperscript{R} ring is enough to prevent escape of small droplets generated by bubbling. When this bubbling diminishes, the alcohol is picked up and replaced by a new fresh portion. This cycle is repeated till complete dissolution of the lithium, taking care of
a possible attack of the copper support by the lithium hydroxide. All the alcoholic fractions are collected in a quartz beaker, in presence of 0.5ml of distilled nitric acid, to prevent hydrolysis of beryllium in presence of LiOH. After evaporation by infra-red heating of the alcohol, all traces of its, which can wash out HDEHP from the column, are eliminated by three successive evaporations to dryness of concentrated HN03. Finally the residue is dissolved in 35 ml of 5.10^{-3}M HNO3, to have a low lithium concentration.

5 Chemical separations.

The first step is the elimination of the bulk of lithium, based on the difference of extrabilities of Li and Be by HDEHP from 5.10^{-3} M HN03 solution. The percolation of the previous nitric solution, at a low flow rate of 1 ml per 5 minutes, fixes the ^7Be on the column when Li passes through. The effluent is collected by 5 ml fractions, the low activity of each fraction being controlled. After percolation of the initial solution, the beaker is decontaminated three times with 2 ml of 5. 10^{-3} M HN03 and finally the HDEHP column is washed by two times 5 ml of pure 5.10^{-3} M HN03. Better than the slow elution with 4M HN03, due certainly to Be hydrolysis, the elution with 1 M hydrofluorhydric acid permits to recover more than 90% of the ^7Be activity in the first milliliter, 95% in two milliliters and 98% in four milliliters, important to minimize the introduction of impurities in the ^7Be fraction. The second purification on a cationic exchanger resin is necessary to eliminate traces of HDEHP washed out with HF. So the different HF fractions are successively evaporated in a TeflonR cones. To eliminate HF traces, three times 0.5 ml of concentrated HN03 are evaporated to dryness.

The ^7Be is then dissolved in the cones by five successive additions of 0.4 ml of 0.4 M HN03 and directly picked from the cones to the ^7Be desorption which is realized by 3 ml of 2M HN03 collected directly in a new TeflonR cone.

The evaporation to dryness, the pick-up of the ^7Be by 3 times 0. 1 ml of 8M HCl and the flow of this solution through the small AGMP1 column ensure complete elimination of iron impurities observed in the previous tries despite all precautions.

The yield of all these separation processes is greater than 90%
6  $^7$Be target.

The preparation of an uniform thin deposit of a high yield of $^7$Be on a thick platinum disk($\phi = 20\text{mm}$, $e = 0.2 \text{mm}$) was developed by molecular plating of $^7$Be nitrate which was dissolved in 2-methyl- 1 propanol, 99.5% HPLC grade (Sigma-Aldrich Company).

So, after addition of 0.4 $\mu$g of $^9$Be carrier (from beryllium atomic absorption standard solution, Aldrich Chemical Company, Inc.), the 8M HCl solution of $^7$Be is evaporated to dryness in a new Teflon cone. The chloride is converted into nitrate by two successive evaporations of 0.2ml of distilled nitric acid. Finally the Be is picked up by three times 6 micro-liters of $10^{-1}$M HN03 mixed with 1.2 ml of 2-methyl-1 propanol.

This solution is transfered into the electrolytic cell, presented in fig. 1. A Teflon$^R$ ring defines the volume of the cell and the diameter of the deposit ($\phi = 8 \text{mm}$). The two parallel platinum electrodes are strongly pressed on this ring, to prevent leakage, by two metallic pieces protected by Afcodur$^R$ insulators. After 45 minutes of electrolysis, with a 740V applied voltage and a 5 mA current, more than 95% of the $^7$Be activity is deposited. After settling of the solution, the platinum disk is recovered and flamed to red color, to obtain beryllium oxide. The total yield of the operation, starting from the beginning is about 85%.

Two $^7$Be targets were prepared in this manner: the first one had an activity of 26.9 mCi and the second one of 131.7 mCi. In both cases the deposit surface was about 0.5 cm$^2$.

References

[1] F. Hammache et al., Phys. Rev. Lett. 80, 928 (1998); F. Hammache, Thesis, (1999), [http://www-csnsm.in2p3.fr/astromc/](http://www-csnsm.in2p3.fr/astromc/)
Figure 1 Sketch of the device for the electrolysis