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Preparation of isotopically enriched mercury sulphide targets

The primary difficulty in performing nuclear reactions on mercury is to obtain a suitable target. The utilization of amalgam targets has been reported in early publications [1,2]. These targets, however, were lacking homogeneity and in-beam stability. A thorough investigation of literature shows, that HgS, because of its comparatively high chemical and mechanical stability, is one of the more adequate Hg compunds for accelerator target applications. In this presentation we describe the production of HgS targets consisting of an enriched Hg isotope and S of natural isotopic abundance, starting up from HgO. Following the outline given in [3], in this special case HgS can be prepared by dissolving HgO in diluted HNO_3 and subsequent precipitation of the black HgS modification with gaseaous H_2S. The procedure includes the following steps:

1) solving the HgO powder in a nitric acid HNO_3, in a relevant glass test-tube,

2) bubbling a hydrogen sulphide H_2S gas through the above solution. In this step, the HgS compound is precipitating from the solution, in a form of black flakes,

3) centrifuging the test-tube, in order to collect HgS on the bottom of it,

4) removing the acid from the tube by means of a pipette,

5) adding distilled water to the tube in order to rinse the acid from the HgS compound, by means of stirring the deposited compound in water with a relevant stirring stick,

6) centrifuging again the tube, removing water (pipette),

7) the procedure of rinsing the acid away, described in the points 5) and 6), is repeated 5 times,

8) rinsing with water the HgS out of the test-tube onto a flat glass saucer,

9) removing an excess of water with a pipette,

10) drying the HgS entirely under an infra-red lamp.

The H_2S gas is supplied from a small lecture bottle and exits the bottle via a regulation valve. Then it is supplied by means of the Teflon tube (10 mm diameter) into a hermetically closed glass wash bottle, serving as an intermediate collection buffer and further pressure reduction stage for the gas. This bottle is connected with the same type of tube to another bottle, in which the gas-supplying tube is immersed in a distilled water, filling the bottom part of the bottle. Subsequently the gas flows out of the bottle via Teflon tube, at the end of which a short glass tube is inserted. The latter tube is immersed in the test-tube containing the nitric acid, in which the mercury oxide is solved. The second bottle is needed in order to stop a possible backflow of the acid from the test-tube towards the gas bottle. In this set-up, the H_2S gas was carefully bubbled through the HgO solution for 30-60 s, until no more black flakes (HgS) precipitated from the solution. Afterwards the test-tube was centrifuged (see p. 3)).

In our case, a desired mercury isotope was ^{204}Hg (90% enrichment), quantity 60 mg, initially in the oxide form.

Before converting it to HgS, we have performed several tests with natural mercury oxide ^{nat}HgO. In these tests we have determined an overall efficiency of the converting procedure, which amounted to 90-95% (determined by weighing the initial mercury oxide and resulting sulphide powders). This is an important improvement in comparison to 65% obtained before with a ball mill [3]. Subsequent conversion of ^{204}Hg oxide to sulphide was performed, to minimize handling risk and avoid an accidental loss of the whole material, in 3 separate batches, in each of them starting with 20 mg of mercury.

Last step of the target production procedure, evaporation of HgS in vacuum, was described in our previous paper [3]. In the present case, the HgS layer of 500 μg/cm^2 on a backing carbon foil of 26 μg/cm^2 with a protective carbon layer of about 20 μg/cm^2 thickness on top of the HgS layer were produced.

Preparation of ^{231}Pa targets

^{231}Pa is an alpha-emitter belonging to the actinium radioactive series. It has a half life of $3.276 \cdot 10^4$ a, corresponding to a specific activity of 1.75 MBq/mg. Being in equilibrium with a series of highly gamma-emitting daughters, it shows a comparatively high gamma dose rate. For example, an unshielded sample of 50 mg produces a doserate of 1 mSv/h at a distance of 5 cm. Therefore, adequate radiation protection measures were taken during target fabrication.

The isotopic material was available in the chemical form of brown coloured, dried nitrate. The brown colour results from radiolytic decomposition of the originally white $PaO(NO_3)_3 \cdot nH_2O$. Since the most favourable source material for actinde target production is the oxide form [4], the Pa nitrate had to be converted to Pa oxide. This was performed by heat treatment of the nitrate in open air in a small electric furnace. The reaction was conducted at a temperature of 673 K in a quartz crucible. After a period of 2 hours, the brown Pa nitrate was quantitatively converted to the white oxide form Pa_2O_5.

Targets of thickness 70-100 μg/cm^2 were produced by cold crucible electron beam evaporation-condensation [4,5] on thin carbon backings of 20 μg/cm^2 as well as on 0.2 mm thin sheet aluminum and on 1.5 mg/cm^2 aluminum foil. The target spots had diameters between 5 and 8 mm and showed mauve to blue-green interference colours, depending on the thickness, and indicating good homogeneity of the condensed layers. A target spot of 8 mm diameter and 100 μg/cm^2 has an activity of 87.5 kBq.

References

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