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Techniques for purification of MoO_3 for AMoRE experiment

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The effectiveness of the purification of molybdenum tri-oxide MoO_3 powder by combination of double sublimation under low vacuum and wet chemistry technique (co-precipitation followed by complete precipitation of poly-ammonium molybdate in acidic media) as well as precise ICP-MS method analysis for the obtained ultra-pure MoO_3 product are discussed in this report. Purified MoO_3 powder is used for further growth of molybdenum-based single scintillating crystals for the AMoRE (Advanced Mo-based Rare Process Experiment) project which is searching for neutrinoless double beta decay ($0\nu\beta\beta$) of ^{100}Mo.

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1.Introduction

For all the molybdenum-based crystals being prepared for the AMoRE experiment, MoO_3 powder is used as the main initial material for crystal growth. Enriched $100MoO_3$ powder (99.997% purity) manufactured by a centrifugal method with about 100 ppt of total Th and U contamination is quite expensive, while usage of commercially available natural 99.95% MoO_3 of ppb-level impurities could help for understanding of purification mechanism and investigation of techniques for the purification of MoO_3. To conform the effectiveness of purification and purity level of obtained product, the concentrations of Th, U and other impurities were checked by ICP-MS analysis (Agilent 7900, USA).

2.Sublimation

Molybdenum trioxide is volatile at temperatures below the melting point. Under low vacuum (<10 mTorr) and in a temperature range of 700-720 °C MoO_3 evaporates without melting and only leaving non-volatile impurities behind. After a successive double sublimation of 1.5 kg raw MoO_3, Th and U contaminations significantly reduced below 20 ppt and 130 ppt levels, respectively, as well as many other contaminants (Sr, Ba, Al, Fe, Mg, etc.) except Pb which has its evaporation temperature of about 400 °C.

3.Wet chemistry technique

Combination of the sublimation and a wet chemistry techniques like co-precipitation with $CaCl_2$-agent followed by complete precipitation of polyammonium molybdates in acidic media provides deep removal of partially remaining radio impurities after the sublimation and allows to produce a uniformly fine powder with Th and U concentrations below 10 ppt.

4.ICP-MS analysis

To confirm the purity level of obtained product and derive the decontamination factors for the reduction of impurities, an ICP-MS analysis using the standard addition method in UHMI and DRC modes was used. For the sample preparation, microwave digestion with mixture of nitric and hydrochloric acids were applied. The detection limit for Th and U using the above method is 10 ppt, moreover, the method shows accurate and stable measurements despite high matrix effect of analyte.

References

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