## §18. Removal of Metal Impurities from Flibe by Electrolytic Separation

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Flibe is a candidate tritium breeding materials in the design of fusion blanket systems. This selection is based on its low MHD pressure loss, low reactivity with air, low pressure operation, and low tritium solubility. Thus, Flibe has been adopted in some self-cooled liquid blanket designs such as the FFHR2. The purification of Flibe is one of the key issues. The advantage of electrolytic purification is that the concentration of impurities can be roughly detected during the purification and at the same time, the removal of metal impurities can be readily carried out. The goal of this work was to remove impurities by electrolysis, a method that can provide in-situ purification and also quantitative measurement of impurity removed.

A schematic drawing of the experimental system is shown in Fig. 1. Using a Be rod or Pt attached to a Ni tube accompanied with flowing H<sub>2</sub> gas as reducer, metal impurities were deposited on a Ni plate electrode. A reference electrode of Pt wire was correlated each hour by referring to the Be<sup>2+</sup>/Be equilibrium potential originated from deposition and dissolution of the solvent. Cyclic voltammetry was carried out with (a) reagent grade Flibe, (b) Flibe with 0.4 wt % NiF<sub>2</sub> impurity and (c) Flibe with 0.4 wt % FeF<sub>3</sub> impurity to measure the electric current of impurity deposition. Impurity metals in Flibe were deposit on the working electrode by chronopotentiometry after each cyclic voltammetry run. The current was controlled to 50 mA.

Cyclic voltammograms in Flibe with NiF<sub>2</sub> and FeF<sub>3</sub> impurities and in reagent grade Flibe are shown in Fig.2. The deposition and dissolution equilibrium was monitored at a potential of  $\pm 0.04$  V from the reported values. As the initial potential of the solvent before scanning was 0.05V, most of the Fe in Flibe is surmised to be reduced and present as Fe<sup>2+</sup>, which is in agreement with equilibrium in the cyclic voltammogram. As the reduction current of each impurity was monitored during the relatively high scanning speed of 25 mV/s and no other reduction reactions were found in the potential window of the deposition, it was inferred that deposition of impurities is possible over a wide range of potential, estimated to be -1.58 V to -0.03 V with only a slight error in the reference potential.

Electrolysis using counter electrodes composed of a Be rod or Pt attached to a Ni tube which was accompanied by flowing H<sub>2</sub> gas was used to separate metal impurities contained in Flibe. The deposition of metal impurities from reagent Flibe was carried out grade with chronopotentiometry at a current of 50 mA for 20 hours using the plate electrode. With a current density of  $0.65\pm0.13$  mA/mm<sup>2</sup>, chronopotentiometry was carried out and metal impurities were deposited on a Ni plate electrode. Enrichment ratio compared to raw materials was 29 for Fe and  $5.2 \times 10^4$  for Ni, which is comparable to results obtained by the combined gas method. The low enrichment ratio of Fe is believed to be due to the detachment of deposit Fe from the deposition electrode. Current efficiency was 14.9% for Fe, Ni and Cr separation.

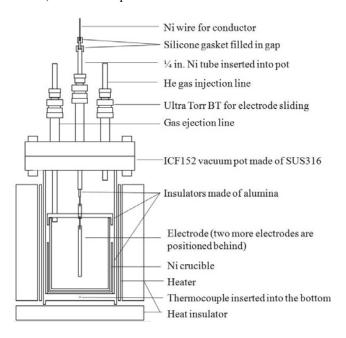


Fig. 1 CAD image of experimental system

