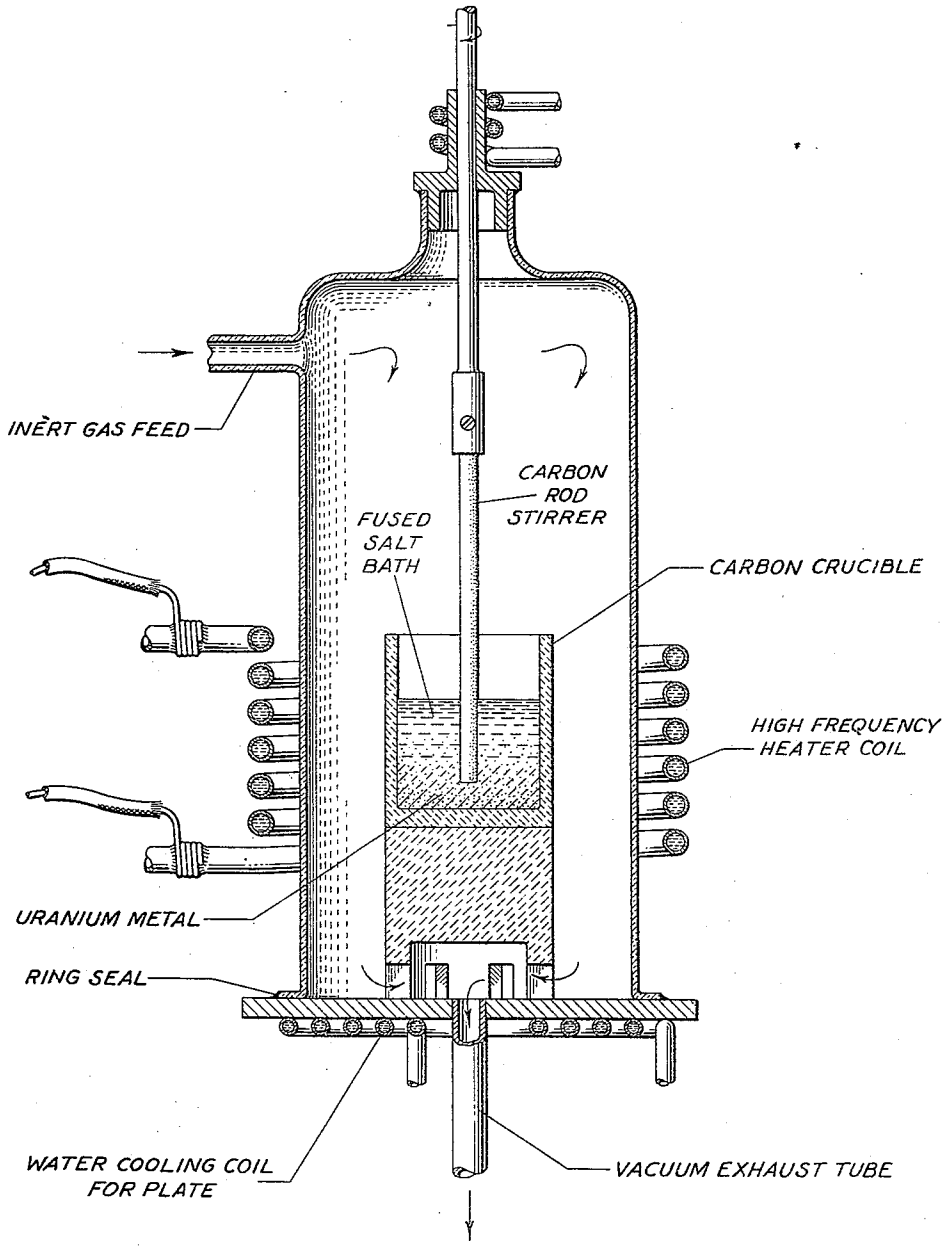
Oct. 9, 1956 G. MEISTER 2,766,110

METHOD OF REFINING URANIUM

Filed March 8, 1944



INVENTOR.
GEORGE MEISTER

BY

Roland A. Anderson

ATTORNEY.

United States Patent Office

2,766,110

Patented Oct. 9, 1956

2,766,110

METHOD OF REFINING URANIUM

George Meister, Newark, N. J., assignor to the United States of America as represented by the United States Atomic Energy Commission

Application March 8, 1944, Serial No. 525,601

3 Claims. (Cl. 75—84.1)

This invention relates to the refining of uranium and more particularly to a method of refining and purifying uranium powder produced by electrolysis or produced by other reduction processes.

Methods and apparatus for the recovery of uranium by the electrolysis of uranium compounds in a fused salt bath are disclosed in copending application Serial No. 478,270, filed March 6, 1943, by William C. Lilliendahl et al., now United States Letters Patent No. 2,690,421, issued September 28, 1954. Briefly, the method thereof includes the electrolysis of a uranium halide in a fused salt bath in which uranium is deposited on a cathode in the form of a powder, the refining of the uranium powder to free it of soluble impurities by grinding and washing operations, and finally the compression of the uranium powder into a slug or button. The present invention relates to improvements in methods of refining uranium, particularly electrolytically deposited uranium powder, such as the uranium deposits produced by the methods and apparatus of said application.

Uranium metal prepared by electrodeposition from a fused salt bath is generally in the form of powder containing more or less impurities including uranium oxide, metals, and salts from the fused bath. As set forth in the above-mentioned application, such powdered uranium may be refined and purified by a series of crushing, washing, grinding, drying, sintering and fusing operations to obtain a body of coalescent uranium suitable for commercial uses.

The present invention discloses that impure uranium may be refined and purified by treatment at high temperatures in a fused salt bath to remove oxides, salts and any other impurities. An alkali acid fluoride bath, such as potassium acid fluoride is found to be particularly effective.

It is the object of this invention to provide a method of refining and purifying uranium by separating the impurities from the uranium while simultaneously fusing the pure metal so produced.

It is an object of this invention to provide a method of fusing uranium powder to obtain uranium in coalescent form.

It is another object of this invention to provide a method using a fused salt bath for purifying and fusing uranium powder.

It is still another object of this invention to provide a fused salt bath for refining impure uranium at temperatures above the melting point of uranium.

Other objects and advantages of the invention will be readily apparent to those skilled in the art from the following description of preferred methods and bath compositions embodying the invention.

The drawing is illustrative of apparatus which may be employed in practicing the method which constitutes the subject matter of the present invention.

According to the method of the present invention, uranium powder, such as that produced electrolytically

in accordance with the disclosure of said copending application, is refined in a fused salt bath at temperatures above the melting point of uranium. The uranium to be refined may be the electrodeposited powder, as mentioned above, or it may be uranium produced by other methods and/or uranium partially refined by crushing, washing, sintering, fusing or similar operations. The fused salt bath may include sodium chloride, NaCl, calcium chloride, $CaCl_2$, barium chloride, $BaCl_2$, sodium fluoride, NaF, calcium fluoride, CaF_2, barium fluoride, BaF_2, or magnesium fluoride, MgF_2, and the like, alone or in combination. A particularly suitable bath contains a potassium acid fluoride, such as KHF_2, in combination with another halide, such as barium chloride, $BaCl_2$. The ratio of the acid fluoride to the halide is not critical except that the mixture should be such as to be fused and remain molten at temperatures of the order of 1000° to 1200° C. in order to be molten at temperatures above the melting point of uranium metal. An acid fluoride bath appears to be more satisfactory than a straight fluoride bath, possibly because of better heating and because the acid fluoride more readily dissolves the oxide impurities. The temperature of the bath should be maintained at about 1100° to 1200° C. during the refining operation by any suitable means such as by resistance heating or by high frequency induction heating in the event that the heat capacity of the bath is insufficient to maintain a temperature above the melting point of uranium long enough for the impurities to be separated from the uranium.

The preferred method of this invention includes the steps of heating the mixture of salts until a fused bath having a temperature of 1000° to 1100° C. is attained, adding the impure uranium to be refined to the fused bath, raising the temperature of the fused mixture to approximately 1200° C., and maintaining said temperature for about ten minutes while stirring the mixture with a carbon rod. Similar methods, such as adding the fused salts to the impure uranium, or fusing the salts and uranium together in an inert gas or in vacuum, may also be used. The described treatment apparently refines the impure uranium by vaporizing certain impurities and by converting other impurities into products which vaporize and/or products which dissolve or remain in the bath mixture. While the refining operation may be conducted successfully in air, the presence of a circulated inert gas or a vacuum is advantageous to carry away the volatile impurities including volatile fluorides. The particles of uranium fuse and coalesce in the salt bath to form pure uranium in coalescent form. The fused uranium may be allowed to solidify in the bottom of the crucible under a layer of slag and a layer of salt, and may be subsequently recovered by breaking open the crucible. The fused uranium may also be recovered by being tapped from the bottom of the crucible and cast in a suitable mold or molds.

By use of the methods and compositions set forth above, uranium in either the powder form or a dense form may be easily refined to produce fused uranium in coalescent condition free of oxide and salt impurities and free of metals such as metallic sodium. The novel method disclosed is particularly economical since it eliminates crushing, washing, grinding, sintering, and similar steps, and eliminates the losses inherent in such steps.

Various changes and substitutions may be made in the methods and compositions described without departing from the spirit and scope of my invention as defined in the following claims.

I claim:

1. The method of refining uranium which comprises the steps of preparing a fused bath containing essentially a potassium acid fluoride and barium chloride, heating

said bath to a temperature of approximately 1100° C. to 1200° C., adding impure uranium to said fused bath, and collecting substantially pure uranium in coalescent form under said bath.

2. The method of refining uranium which comprises the steps of preparing a fused bath containing essentially a mixture of a potassium acid fluoride and barium chloride, heating said bath to a temperature of about 1100° to 1200° C., adding impure uranium powder produced by electrolysis to said fused bath while maintaining a vacuum over the surface of said bath, and collecting substantially pure uranium in coalescent form under said bath.

3. The method of refining uranium which comprises the steps of preparing a fused bath containing essentially a mixture of a potassium acid fluoride and barium chloride, heating said bath to a temperature of about 1100° to 1200° C., adding impure uranium powder produced by electrolysis to said bath while maintaining a flow of an inert gas over the surface of said bath, and collecting substantially pure uranium in coalescent form under said bath.

References Cited in the file of this patent

UNITED STATES PATENTS

1,180,435	Robison	Apr. 25, 1916
1,568,685	Moore	Jan. 5, 1926
1,814,721	Marden	July 14, 1931
1,841,599	Hardesty	Jan. 19, 1932
1,861,625	Driggs et al.	June 7, 1932
1,968,984	Binder	Aug. 7, 1934
2,170,863	Junker et al.	Aug. 29, 1939
2,261,905	Nelson et al.	Nov. 4, 1941

FOREIGN PATENTS

230,865	Great Britain	Dec. 10, 1925

OTHER REFERENCES

Goggin, Cronin, Fogg, James: "Industrial and Engineering Chemistry," Feb. 1926, pp. 114–116.

W. Kroll: "Zeitschrift für Metallkunde," Feb. 1936, pp. 30–33.