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TITLE

REPORT OF INVENTION - THE APPLICATION OF FLUIDIZED BEDS TO VOLATILITY SEPARATION PROCESSES - HWIR 462

AUTHOR

M. T. Walling
and
E. E. Voiland

ISSUING FILE

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REPORT OF INVENTION

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TO: M. K. Cain

I: ATTACHED HERETO IS A DESCRIPTION OF WHAT MAY BE AN INVENTION IN: July 27, 1953

Post BNW

The Application of Fluidized Beds to Volatility Separation Processes

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II: THE NAME, TITLE OR POSITION, WORKS LOCATION, AND PERMANENT ADDRESS OF THE INVENTOR(S) IS:

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E. E. Voiland, Chemist, Hanford Atomic Products Operation,
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III: EVIDENCE AS TO WHEN AND WHERE THE INVENTION WAS MADE CAN BE FOUND IN THE FOLLOWING LISTED WRITTEN OR PICTORIAL MATERIAL (NOTEBOOK, FILE REPORTS OR DRAWINGS, ETC.):

Bushey, A.H., Quarterly Progress Report, Chemistry Unit, April, May, June, 1953
HW-28634, 7/3/53, p. 65.

[redacted]

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IV: THE APPROXIMATE DATE OF THE FIRST ENTRY ... WRITTEN ... MATERIAL DESCRIBING OR SHOWING SAID IN... IS:

July 3, 1953

APPROVED FOR PUBLIC RELEASE 11/17/97

V: PERSONS WHO COULD TESTIFY AS TO WHEN AND WHERE THE INVENTION WAS MADE INCLUDE THE FOLLOWING:

H. H. Hopkins, Jr.
R. G. Post

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BY ... DATE 6-4-81
BY S W Jordan DATE 6-8-81

THE APPLICATION OF FLUIDIZED BEDS TO VOLATILITY SEPARATION PROCESSES

Fluidized solid beds possess certain attributes which may make them useful in connection with the problem of decontaminating and purifying uranium by distillation or sublimation of a volatile uranium compound. These include:

(1) High gas-solid heat transfer.
(2) Solids may be easily and continuously added to or withdrawn from the fluidized bed.
(3) The entire solid surface is exposed to the fluidizing gas stream, resulting in high rates of reaction of the solids with the gas.
(4) The bed solids may be moved in the fluidized state by techniques very similar to ordinary gas moving methods.

Possible applications of fluidized beds to the decontamination of uranium are illustrated for the preparation and purification of uranium hexafluoride from Hanford slugs in the sections following.

Process A

(1) Decanning of slugs by current techniques.
(2) Dissolution of decanned slugs in nitric acid.
(3) Separation of plutonium by solvent extraction (e.g., with thenoyl trifluoroacetone, TTA), precipitation, or other suitable technique. The uranium is retained in the nitric acid solution in this step.
(4)* Evaporation of the aqueous uranyl nitrate solution to a concentrated solution, e.g., a uranyl nitrate hexahydrate "melt", which is fed continuously to a fluidized bed of uranium trioxide. This bed is heated externally and the fluidizing gases are preheated before entering the bed. Evaporation of the residual water and de-nitration to uranium trioxide are accomplished in this bed.
(5) Uranium trioxide formed in Step 4 is fed continuously to a fluidized, heated bed of uranium dioxide. The fluidizing gas fed to this bed contains hydrogen or ammonia or other suitable gaseous reducing agent. The uranium trioxide fed to this bed is converted to uranium dioxide by this treatment.
(6) The uranium dioxide formed in Step 5 is fed continuously to a fluidized, heated bed of uranium tetrafluoride. The fluidizing gas fed to this bed contains hydrogen fluoride or other suitable fluorinating agent which converts the uranium dioxide to uranium tetrafluoride.
(7) The uranium tetrafluoride is fed continuously either to a fluidized bed containing an inert inorganic fluoride or to a simple "combustion chamber". A gas containing gaseous fluorine, gaseous chlorine trifluoride or other suitable fluorinating agent is fed to this unit. The entering uranium tetrafluoride is converted to uranium hexafluoride by this treatment. Pressures and temperatures are so adjusted that the uranium hexafluoride sublimes as it forms. If simple sublimation suffices to give the desired decontamination, the uranium hexafluoride sublimate is simply collected and packaged. If additional decontamination is desired, Step 8 is performed.

*Investigations exploring the possibilities for utilization of fluidized beds to convert aqueous uranyl nitrate solutions to uranium trioxide are currently in progress in the Chemical Development Unit.

(8) The uranium hexafluoride vapor is collected in a suitable solvent (e.g., a perfluorocarbon compound or a suitable inert liquid inorganic fluoride). This solution is then fed to a fractionating tower from which low boiling contaminants are removed as the overhead product, while the uranium-bearing solution is recovered as the bottoms. This uranium-containing solution is fed to a second fractionator from which purified uranium hexafluoride is recovered as the overhead product and the solvent recovered as the bottoms. The solvent is recycled to the uranium hexafluoride scrubber. Periodic or continuous removal of fission products from the solvent may be necessary.

(9) The plutonium removed in Step 3 is purified by solvent extraction, precipitation, or other suitable techniques.

Process B

(1) Decanning of slugs by suitable techniques.

(2) The decanned slugs are treated with a suitable gaseous agent which converts the massive slugs to a finely divided powdered uranium compound, suitable for fluidization. Examples of agents which might be used for this purpose include steam, air, or oxygen.

(3) The finely divided uranium compound formed in (2) is fed to a heated, fluidized bed containing uranium tetrafluoride. The fluidizing gas to this bed contains hydrogen fluoride or other suitable fluorinating agent. The uranium compound is converted to uranium tetrafluoride by this treatment.

(4) The uranium tetrafluoride formed in Step 3 is fed continuously to a fluidized bed containing an inert diluent fluoride. Gaseous fluorine, gaseous chlorine trifluoride, or other suitable fluorinating agent is fed in the fluidizing gas to this bed. The uranium tetrafluoride is converted to uranium hexafluoride by this treatment. Hopefully, the non-volatile fission product fluorides and plutonium fluorides will be retained on the diluent fluoride making up the bed. The diluent fluoride with such fission products and plutonium as are retained on it is removed continuously from the bed and replenished with fresh material. Pressures and temperatures are so adjusted that the uranium hexafluoride sublimes continuously as it forms.

(5) The uranium hexafluoride vapor is collected in a suitable solvent. The resultant solution is fed to a fractionating tower where low boiling contaminants are removed as the overhead product while the uranium-containing solution is recovered as the bottoms. This uranium-containing solution is fed to a second fractionator from which purified uranium hexafluoride is recovered as the overhead product and the solvent recovered as the bottoms. Any non-volatile fission product fluorides or plutonium fluorides will, hopefully, slurry with the solvent and will be collected with the bottoms. Any volatile plutonium hexafluoride formed in (4) may have to be removed from the product uranium hexafluoride stream by scrubbing with a mild reducing agent, e.g., a Freon.

(6) Plutonium collected on the diluent bed material and in the solvent in the fractionator will be combined and processed in an as yet unknown fashion. (Probably by dissolution and solvent extraction.) The solvent will be recycled to the uranium hexafluoride scrubber.

Process C

(1) Decanning of slugs by current techniques.
(2) Dissolution of the decanned slugs in a suitable medium.
(3) Separation of plutonium from the slug solution by solvent extraction or other suitable technique. The separated plutonium will be purified by solvent extraction or other suitable technique.
(4) Precipitation of the uranium from the slug solution as a filterable uranium tetrafluoride or suitable double salt, e.g., ammonium fluoride-uranium tetrafluoride, $NH_4F \cdot UF_4$.
(5) Centrifugation or filtration of the uranium-bearing precipitate. This precipitate is slurried continuously into a fluidized, heated bed of uranium tetrafluoride. Drying and thermal decomposition to uranium tetrafluoride, if necessary, is accomplished in this bed.
(6) The uranium tetrafluoride resulting from Step 5 is fed continuously to a fluidized bed containing a suitable inert diluent fluoride. Gaseous fluorine, gaseous chlorine trifluoride, or other suitable fluorinating agent is fed in the fluidizing gas to this bed. Uranium hexafluoride is formed in this unit and sublimes as it forms.
(7) If necessary for decontamination, the uranium hexafluoride vapor resulting from (6) is scrubbed out into a suitable solvent. This solution is fed continuously to a fractionator from which low boiling contaminants are removed as the overhead product and the uranium-containing solution removed as the bottoms. This uranium-containing solution is fed to a second fractionator from which purified uranium hexafluoride is recovered as the overhead product and the solvent recovered as the bottoms. The solvent, after whatever purification is necessary, is recycled to the uranium hexafluoride scrubber.

It is the authors' belief that Steps A(5), A(6), A(7), B(3), B(4), C(5), and C(6) of the foregoing descriptions represent new applications of fluidized beds. Step A(4) does not represent a new application, and the authors make no claim to having originated this idea.

Matthew Taylor Walling, Jr.
Inventor

Date July 24, 1953

Eugene E. Voiland
Inventor

Date July 24, 1953

Horace H Hopkins, Jr. 7-24-53
Witness Date

Roy G. Post 7-27-53
Witness Date