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TITLE
REPORT OF INVENTION - THE SUPPRESSION OF FLUORIDE VOLATILIZATION IN NITRIC ACID DISTILLATION

AUTHOR
R. L. MOORE

NORTHWEST LABORATORY
RECEIVED
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1-6. WC Poe
7. WH Reas
8. MT Walling, Jr.
9. RL Moore
→10-12. TID - Oak Ridge
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ATOMIC PRODUCTS DIVISION
GENERAL ELECTRIC COMPANY
RICHLAND, WASHINGTON

A.E.C. CASE NO.

G. E. CASE NO.

REPORT OF INVENTION

TO: W. C. Poe

I: ATTACHED HERETO IS A DESCRIPTION OF WHAT MAY BE AN INVENTION IN:

The Suppression of Fluoride Volatilization in Nitric Acid Distillation.

Classification Cancelled And Changed To
DECLASSIFIED -UNCLASSIFIED
By Authority of TID-1381
Suppl. 12-31-71.
By B Roberts 6/22/88
Verified By B. Cleary 6-22-88

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

Robert Lee Moore, Head - Chemical Research
Hanford Atomic Products Operation
310 Adams, Richland, Washington

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.):

Notebook HWN-1103, p. 50, November 1, 1955.

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

November 1, 1955

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BY AUTHORITY OF CCRS
BY JR DATE 5-26-76

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

M. T. Walling, Jr.
J. L. Hepworth

SPECIAL RE-REVIEW
FINAL DETERMINATION
DECLASSIFICATION CONFIRMED
BY AE Baker DATE 6-28-82
BY [illegible] DATE 6/28/82

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SIGNED (SUPERVISOR)	DATE	DEPARTMENT
M. T. Walling, Jr.	7/25/56	ENGINEERING

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NOTE: SUGGESTIONS FOR PREPARING THE INVENTION DESCRIPTION ARE CONTAINED ON THE REVERSE SIDE OF THIS FORM.

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DESCRIPTION OF INVENTION

Many of the proposed reactor fuel elements incorporate passive fuel alloys or jackets which will require at least catalytic amounts of fluoride to bring about their dissolution in nitric acid. Studies of the behavior of this fluoride in the Purex acid recovery process(1)(2)(3)(4)(5) indicate that it will reflux and build up in the Purex acid fractionator and thus, because of the attendant corrosion problems, constitute the major obstacle to the processing of such fuel elements in the Purex plant. To prevent this difficulty, it is proposed that a small concentration of an inorganic fluoride complexant, such as zirconium(IV), aluminum(III), thorium(IV), or iron(III) be added to the fluoride containing solutions. This will prevent the fluoride from volatilizing with the recovered acid and concentrating in the acid fractionators, or being recycled to process. Instead it will be discharged in the IWW waste stream. Use of the fluoride complexing agent will have the further benefit of drastically reducing corrosion of both the acid fractionator pot and of the waste storage tanks. Experimental data supporting these claims is presented in the following paragraphs.

The distribution coefficient of fluoride between boiling 8 M nitric acid (the concentration in the waste fractionator bottoms) and its equilibrium vapor is shown in Table I as a function of type and concentration of additive (all added as the nitrate salts).

TABLE I

EFFECT OF COMPLEXING AGENTS ON FLUORIDE DISTILLATION

All solutions 8 M HNO_3, 0.1 M F^-

Additive	M Additive	K (HF)*	Volatility Depression (R)
None	--	0.78	--
Zr(IV)	0.025	0.28	2.8
Zr(IV)	0.05	0.067	11.6
Zr(IV)	0.1	<0.006	>130
Al(III)	0.05	0.25	3.1
Al(III)	0.1	0.091	8.6
Al(III)	0.25	0.0183	43
Th(IV)	0.1	0.08	10
Fe(III)	0.25	0.21	3.7

* K(HF) = ratio of concentration of fluoride in equilibrium condensate to that in still bottoms.

It will be noted that, of the complexing agents tested, zirconium was by far the most effective. In fact 0.1 molar zirconium (added as zirconyl nitrate) reduced the fluoride concentration in the distillate to less than the analytical detection limit (0.01 grams fluoride per liter). Aluminum nitrate was also effective, although less so than zirconium. Thorium nitrate appeared to be about equal to aluminum on a molar basis. However, in this case a precipitate tended to form in the still pot on cooling. Complexing with iron was much weaker than with the others, and a large molar excess would be required to achieve the same reduction in fluoride volatilization.

It was noted that corrosion of the 309 SCb stainless steel still was negligible and that the solutions remained water-white when aluminum, thorium, or zirconium was present. In the absence of these complexants, corrosion was severe, copious evolution of oxides of nitrogen (indicating attack of the metal) was observed, and the pot solution took on the color of the dissolved chromium and iron corrosion products. Microscopic examination of test coupons showed a pitting type attack in the nitric acid - hydrofluoric acid solutions which was not observed when the fluoride complexants were present.

Additional experimental data, as well as information on the equilibria in the nitric acid - water - trace fluoride system, is given in the following reports:

(1) HW-40692-H, Separations Section Monthly Report - December, 1955, p. 40.

(2) HW-41205-H, Separations Section Monthly Report - January, 1956, p. 50.

(3) HW-41702-H, Separations Section Monthly Report - February, 1956, p. 43, 44.

(4) HW-42219-H, Separations Section Monthly Report - March, 1956, p. 69.

(5) HW-42626-H, Separations Section Monthly Report - April, 1956, p. 51.

Robert L Moore 7/25/56
Inventor Date

Read and understood by me this 30 day of July, 1956.

Matthew T. Walling, Jr.
Witness

John L. Hepworth
Witness

RL Moore:rd

[redacted]