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AEC RESEARCH AND DEVELOPMENT REPORT

PRODUCTION OF (URANIUM) TETRAFLUORIDE

NYO-5001

Harshaw Chemical Company

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For The Atomic Energy Commission
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May 13, 1943

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PRODUCTION OF TETRAFLUORIDE

NYO-5001

The Harshaw Chemical Company
May 13, 1943

Production of tetrafluoride for the month of April was 49,400#. New equipment installed late in March operated smoothly and the production was slightly over the rated capacity of the plant.

Nickel ends were welded on to the steel reactor tubes to prevent corrosion at the cover joint and possible contamination of the product with iron. The nickel ends will also save considerable repair work on the tube ends. The magnesium trays for some of the reactors are beginning to crack at the sides from frequent straightening. The trays are bent out of shape by the expansion of the product and are reshaped by hammering over a die. Some of the cracks have been repaired by welding but this is not very satisfactory because the welded trays are harder to straighten and often crack again.

The Chemical 42-17 used in April was quite uniform in activity, averaging 79% compared with 79% in February and 80% in March. The assays of this material all passed specification except a single drum lot, No. M1323DK97. This drum assayed 95.83%.

The finished product analyzed as follows:

	MF ₄	MO ₂	MO ₂ F ₂	M
April, Average	98.49	.20	1.27	75.83
Low	97.01	.06	.69	75.78
High	99.22	.45	2.70	76.02

An improved method of sampling the finished product was introduced during the month which consists of taking a core sample of each drum for compositing. Previously a grab sample was taken from each tray.

The average HF efficiency of the process from the beginning to May 1st has been:

Input	586% of theory
Recovered 30% acid	448% of theory
Acid used up	138% of theory

The input of HF could be lowered by running the reaction for a longer time but by so doing the production would be cut in proportion to the extra time required.

K.E. Long/mk

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The Harshaw Chemical Company
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WATER SOLUBLE NO_2F_2

The NO_2F_2 calculated from the total soluble M corrected for the soluble tetravalent M has proven to be a rapid direct method for analytical control over oxidation from air leakage, etc. in the manufacture of the green salt. Using a standardized leaching procedure reasonably reproducible values are obtained which are more consistent than those for total and tetravalent M. As for absolute values, the method appears to give high results due to a progressive oxidation depending upon time and amount of contact with air during the leaching step. Ordinary distilled water with no air removed gives values two to three times as high as those obtained where air is removed and a nitrogen atmosphere used. Boiled water cooled in a stoppered flask gives values intermediate between these.

There is some indication that 1% - 5% H_2SO_4 leach may give values of the same order as those with the nitrogen atmosphere when considering the difference between total and tetra M.

In order to illustrate the effects of various leaching procedures as to the amount of oxidation and reproducibility of results, the following table gives the values obtained on the same material. All leachings were made on a 5 g. sample at room temperature in 250 ml with different ways of excluding air on sample ML743DT10 which analyzed: Total M 75.81 by ignition method; NO_2 0.10 by oxalate insoluble.

Method of Leaching	Time of Leaching Hrs.	Soluble Tetra-valent M %	Soluble Total M %	Hexa-valent M by Diff. %	MO ₂ F ₂ %
A. Distilled water	1	.31	.86	.55	0.71
boiled and	1	.29	.86	.57	.74
cooled in a	1	.37	.93	.56	.73
stoppered flask.	2	.50	1.17	.67	.86
	2	.56	1.40	.84	1.09
B. Distilled water	1	.12	.53	.41	.52
warmed and	1	.48	.84	.36	.47
evacuated then	1	.20	.74	.54	.69
manipulated	2	.37	.92	.55	.71
under nitrogen.					
C. Distilled water	1	.26	1.12	.86	1.11
with no air	1	.54	1.39	.85	1.10
removed.	2	.74	1.84	1.10	1.42
D. Same as A + 1% H ₂ SO ₄	1	.73	1.22	.49	.63
E. Same as B + 1% H ₂ SO ₄	1	.68	1.11	.43	.56

Variation in the MO₂F₂ affects the calculated MF₄ and total analysis as follows:

MO ₂ F ₄	.5	.7	1.1
MF ₄ calc.	99.39	99.18	98.77
Total Analysis	99.99	99.98	99.97