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For The Atomic Energy Commission

H. R. Cansell
Chief, Declassification Branch

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ABSTRACT

Metallex Process. The addition of 0.03 wt % magnesium to thorium quasi amalgam after dry reduction and prior to washing increased the reduction and recovery yield by 15%, but also increased the porosity and oxide content of the sintered metal. The addition of magnesium before reduction increased the yield only slightly if at all.

There is a definite correlation between the free voidage of the product thorium metal and the metal quality. As the free voidage increases, the oxide, nitrogen, and hydrogen contents and the metal hardness increase. Residual water in the amalgam at the beginning of the sintering step appeared to increase the free voidage, but anhydrous washing technique increased rather than decreased the free voidage. Correlation between free voidage and the pressure used in cold pressing was poor, but slow heating prior to sintering appeared to improve the quality.

Fluorox Process. Hydrogen at 475°C did not remove residual carbon from UF_4 pellets contained in a nickel boat.

The specific surface area of UO_2F_2 was found to be much greater than that of UF_4 . The specific surface area of UF_4 remained essentially unchanged in 5 hr in an inert atmosphere at 800°C.

A compound, which is possibly UF_6 , has been isolated as a product of the reaction of UF_4 with dry oxygen.

A semiautomatic thermobalance was constructed for use in Fluorox studies.

The reduction-hydrofluorination reactor and the oxidation reactor are being rebuilt with the modifications shown in 56-6-100. The cold trap, which has been connected to the off-gas line from the oxidation reactor, has been purged through a hydrolyzer to recover the contained UF_6 as a UO_2F_2 solution.

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Excer Process. In applications of the Excer process to ore processing, UO_2Cl_2 solution containing 225 g/liter was obtained as product of the anion exchange column. The sodium concentration was 0.05 M. Conversion of the sulfate with HCl instead of NaCl gave good results.

The uranium was decontaminated from boron by factors of 104 and > 660 in the anion- and cation-exchange columns, respectively. It therefore appears that the boron detected in Excer products is derived from process equipment or chemicals.

The concentration of the acid used to carry the current in the catholyte did not significantly affect the tap density of the UF_4 produced. The tap density was increased by grinding with a mortar and pestle.

UO_2F_2 was successfully reduced to $\text{UF}_4 \cdot 3/4\text{H}_2\text{O}$ in a polyvinylchloride electrolytic cell. Ten pounds of product with a tap density of 2.6 g/cc was produced. The reduction and current efficiencies were 76 and 52%, respectively. During the first dehydration test 24 lb of $\text{UF}_4 \cdot 3/4\text{H}_2\text{O}$ (3.48% H_2O) was converted to UF_4 with 1.47% residual water. The dehydration was performed at 380°C in a nitrogen atmosphere, with 35 min in the heated zone.

1.0 METALLEX PROCESS

In the Metallex process thorium compounds are reduced to thorium metal by means of sodium amalgam. From the thorium amalgam thus formed, the thorium is separated by filtering and pressing, followed by heating to distill off the mercury and sinter the residual thorium metal.

1.1 Laboratory-scale Studies (O. C. Dean, C. P. Johnston)

Effect of Moisture in ThCl_4 on Yield. In the presence of moisture, sodium amalgam did not significantly reduce powdered, supposedly dry, ThCl_4 . This was demonstrated in two runs in which no thorium metal was obtained although the conditions were believed to be those that usually gave 70-80% yields. Further investigations showed that the ThCl_4 used in the first run had absorbed sufficient moisture to raise its water content from 0.5 to 1.44% even though it had been stored and sealed in a bottle in a dry box. The amalgam used was 3.3 M in sodium. In the second run, the water content of the sublimed ThCl_4 was found to be 0.75%. In this run the amalgam was only 1.0 M in sodium, and it had been stored about 3 months after generation before being used.

In the best reduction and recovery yield obtained to date, 94% of the thorium was recovered in the quasi amalgam when 0.03 wt % magnesium was added to the products in a dry reduction of ThCl_4 by $\text{Na}(\text{Hg})$.

prior to washing, in a attempt to prevent oxidation during the washing. It was thought that magnesium would react preferentially with HCl or water to inhibit the reactions

Analytical results showed a 104% thorium material balance; 10% of the magnesium remained with the quasi amalgam. One sinter produced from this quasi amalgam, in which the pressing was done under argon, had an apparent density of 10.4, bulk density of 9.03, and atmosphere-connected voidage of 11.3%. The residual magnesium content of the metal was 1200 ppm, and the ThO_2 was 3.0%. Both are too high for acceptable metal. Also, the free voidage of the sintered metal appeared to be higher when magnesium was used after reduction and before washing, but not when used before reduction:

<u>Mg</u> <u>(wt %)</u>	<u>When</u> <u>Added</u>	<u>Reduction</u> <u>Yield (%)</u>	<u>Th in</u> <u>Quasi</u> <u>Amalgam</u> <u>(%)</u>	<u>Free Voidage</u> <u>in Sintered</u> <u>Metal (%)</u>	<u>Mg in</u> <u>Sintered</u> <u>Metal</u> <u>(ppm)</u>	<u>HCl-insoluble</u> <u>Material in</u> <u>Sintered</u> <u>Metal (%)</u>
0	--	65.75	50-60	7-25	--	0.5-2.0
0.003	Before reduc- tion	75.7	72.8 ^a	24.4	545	3.7
0.03	Before reduc- tion	81.0	57.0	--	--	--
0.03	After reduc- tion	89.4	89.0	19 ^b	1200	3.0

^a 37% of the thorium was recovered in immediate amalgam filtration and the rest in a second filtration 16 hr later.

^b Average of two values.

The high residual magnesium in the sintered metal points to the necessity for increasing the acid concentration in the aqueous amalgam wash, which is now recommended as 1.0 M HCl.

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Relation Between Metal Quality and Free Voidage. Results of further studies on the relation between the quality of sintered thorium metal and the percentage of free voidage showed that higher oxide, higher nitrogen, and higher hydrogen contents of the sintered metal generally were correlated with higher free voidage values:

<u>Sample</u>	<u>Reduction Temp. (°C)</u>	<u>Free Voidage (%)</u>	<u>ThO₂ (by vacuum fusion) (%)</u>	<u>Hydrogen (ppm)</u>	<u>Nitrogen (ppm)</u>
1	125	7.47	0.54	17	21
2	125	13.20	1.40	54	26
3	125	11.70	1.40	48	63
4	200	37.10	3.63	110	390
5	200	34.60	4.29	150	450
6	200	29.0	2.56	74	270

Pressing the resulting sintered metal to 50% thickness required 1.5 times greater pressure for the higher voidage samples, and resulted in metal 1.65 times as hard.

Effect of Washing Procedure on Metal Quality. Residual water in the amalgam was deleterious to the metal quality, but anhydrous washing technics increased the atmosphere-connected voidage of the sintered metal. Washing with methanol distilled from alkaline pyrogallol to remove free oxygen or with HCl-sparged methanol gave thorium amalgams of finer structure than HCl-water washed amalgams. However, washing the amalgam with and storing it under aqueous 1 N HCl gave the only product with near the desired 0-15% atmosphere-connected voidage (Table 1). If free voidage is affected by the washing procedure, the effect takes place early in the washing routine, i.e., when HCl, either aqueous or alcoholic, first contacts the amalgam, or that methanol from which free oxygen has been removed is as reactive with the amalgam as water.

In another series of three experiments, in addition to those in Table 1, free voidage was about 5% higher when nonaqueous washing procedures were used than it was in products similarly treated but washed with aqueous reagents. In these experiments two of the samples were washed with methanol sparged with anhydrous HCl, followed by several rinses with methanol that had been distilled from alkaline pyrogallol to remove free oxygen. The third sample was washed entirely with the distilled methanol. One sample was cold pressed at 1 tsi under argon,

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Table 1. Effects of Washing Reagents and Drying Procedures Used with Thorium Amalgams on the Free Voidage of the Sintered Metal Product

Preliminary Washing Material and Storage Cover	Storage Time (Weeks)	Subsequent Washing Material	Cold Pressing Pressure (tsi)	Free Voidage (%)
3 N HCl	6	H ₂ O	10	28.0
	6	H ₂ O	0.5	42.9
	6	1% Propylenediamine in H ₂ O	10	49.0
	8	Methanol distilled from alkaline pyrogallol in argon stream	10	48.7
	6	Same	0.5	34.8
	4	Same, then vacuum dried	10*	24.0
	4	Same	10	27.2
	4	Same	0.5	34.5
	12	H ₂ O	0.5	13.5
	12	H ₂ O, then vacuum dried	0.5	7.5
Methanol distilled from alkaline pyrogallol in argon stream	12	H ₂ O, then methanol	0.5	11.7
	1/7	Methanol distilled from alkaline pyrogallol in argon stream	5	39.9
	1/7	Same	5	50.9
HCl-sparged methanol	1/7	Same	1	51.5

*Argon atmosphere during pressing.

the second at 4.3 tsi under argon, and the third at 5 tsi under air. All were vacuum-sintered for 1 hr at 1100°C. The three quasi amalgams appeared to contain very small solid particles, which were difficultly retained on the filter and in the cold-press die. On exposure to air, the samples heated spontaneously. This has been observed in only a few cases with samples washed in aqueous reagents.

Effect of Pressing Pressure on Metal Quality. No significant correlation between slug pressing pressure and free voidage was noted.

Effect of Heating During Sintering on Metal Quality. The quality of sintered thorium compacts was found by the Metallurgy Division to be improved by slow heating prior to vacuum sintering of the cold-pressed thorium compacts. Such heating in the temperature range 90-140°C prior to sintering at 1100°C produced sintered compacts with fewer cracks and a less spongy appearance. This suggests that controlling the rate of mercury evolution in an optimum temperature range may produce better quality metal.

The Metallurgy Division also observed that, when very porous thorium sinters are salvaged by burning them in air, a combustible gas is evolved. This suggests the possibility that the porous sintered metal is reactive at or near room temperature with the moisture of the air, producing thorium hydride. Literature sources state that the rate of formation of thorium hydride is negligible at metal temperatures below 250°C. The combustible gas observation implies that the finely divided metal and perhaps the thorium quasi amalgam may form hydrides rapidly at low temperature.

2.0 FLUOROX PROCESS

2.1 Laboratory-Scale Studies (R. G. Wymer, L. M. Ferris)

Removal of Residual Carbon from UF₄ Pellets. When UO₃ is reduced and hydrofluorinated to UF₄ with starch and HF gas, respectively, there is always some residual carbon in the UF₄. Since hydrogen does not react with UF₄ at temperatures below 950°C, it was tried as a carbon-removal agent at 475°C with a nickel boat and tube. No carbon was removed, showing that the nickel did not act as the catalyst needed for formation of methane. By utilizing nascent hydrogen from cracked NH₃, it may be possible to avoid the use of a catalyst.

UF₄ Surface Area Measurements. The specific surface area of UO₂F₂ was found to be much greater than that of UF₄. Since pure UF₄ was not available, 98% UF₄-2% UO₂F₂ was oxidized for an hour at various temperatures to produce UF₄-UO₂F₂ mixtures of known compositions. It was hoped that the specific surface area of pure UF₄ at the various temperatures could then be obtained by extrapolation of the surface area data

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for UF_4 - UO_2F_2 mixtures. However, the data were inconsistent and nonreproducible. Variations in UF_4 sintering rates, caused by packing the fine powder into a boat differently each time, probably account for most of the inconsistencies found. The specific surface area of UF_4 purified by water leaching of 98% UF_4 -2% UO_2F_2 was 0.06 m^2/g at 25°C and 0.04 m^2/g at 800°C, which is about 1/4 that of the 98% material. Retention times up to 5 hr at 800°C did not appreciably change the surface area of the UF_4 .

Intermediate Compound Formation in $UF_4 \rightarrow UF_6$ Reaction. A compound, which is possibly UF_5 , has been isolated as a product of the reaction of UF_4 with dry O_2 . For some time it has been observed that a brown-colored compound is swept out of the reaction chamber simultaneously with the UF_6 during oxidation. This phenomenon has occurred regardless of the source of the UF_4 . At room temperature in the presence of moisture the brown substance hydrolyzes rapidly to a 1:1 mixture of UO_2F_2 and UF_4 . The brown substance is not very volatile at low temperatures; therefore, UF_6 may be separated from it by volatilization at ~60°C in a stream of dry inert gas. X-ray patterns obtained to date showed a large amount of scatter owing to the use of relatively large quartz capillary tubes. Dry-box loading of the material into the extremely fine capillaries needed for definitive X-ray analysis has proved difficult. However, the pattern obtained indicates that the substance is possibly β - UF_5 . This indication is in agreement with the observed chemical properties of the material, which coincide with the known chemical properties of UF_5 . If, by further investigation, the material is definitely shown to be UF_5 , it will be the first time that this compound has been isolated as a product of the reaction of UF_4 with dry O_2 .

Thermobalance. A semiautomatic recording thermobalance (Fig. 1) was constructed and is currently being used to follow the weight change in the oxidation of UF_4 by dry O_2 . The UF_4 sample is contained in a platinum basket suspended by a 40-gage platinum wire. Any change in weight of the sample is electronically transmitted to a Brown recorder, which continuously plots the weight change vs. time.

Before being used, the thermobalance must be calibrated for buoyancy produced by high gas velocities and other conditions in the flowing gas system. These conditions, in order of magnitude of effect, are: (1) the length of the platinum wire used for suspension, i.e., the distance of the platinum basket from the gas inlet; (2) whether or not the off-gas tube is connected to the NaF trap; (3) the load suspended from the wire; (4) the furnace temperature; and (5) the effect of admitting a slow stream of helium at the top of the reaction vessel while oxygen at a much higher rate is being admitted at the bottom.

With the NaF trap disconnected and using a 19-in. platinum wire (basket is 4-5 in. from the gas inlet), there was an apparent loss of

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Fig. 1 Schematic Diagram of Thermobalance

only 1 mg in the weight of a 30-g platinum basket as the O_2 flow rate increased from 75 to 1500 ml/min. When a 22-in. wire was used, the total apparent weight loss at 1500 ml O_2 /min was about 3 mg.

When the off-gas line was connected to the NaF trap, the buoyant effect on a 30-g load increased to about 12 mg by increasing the O_2 flow rate from 75-1500 ml/min. At gas flow rates up to 300 ml/min there was no apparent buoyant effect on a 30-g load even with the NaF trap connected. When a platinum basket weighing 14 g was used instead of the 30-g basket, the buoyant effect of O_2 at 300 ml/min was very small (0.5 mg). At higher flow rates the apparent weight loss was greater than in the case of the heavier load. For example, at 1200 ml/min the apparent weight loss was 10 mg as compared with 7.5 mg from the 30-g boat. The buoyant effects on an 18.5-g load were almost identical with those for 14 g.

The slow admission of helium at the top of the container while O_2 was being admitted at the bottom had little or no effect on the buoyancy. The furnace temperature had no effect. All weight changes produced by adding or subtracting known weights on the balance were reproducible to within 0.5 mg.

2.2 Larger-Scale Studies (J. C. Bresee)

Reduction-Hydrofluorination. In the rebuilt reduction-hydrofluorination equipment (CF-56-6-100, Figs. 1 and 2) the HF flow to the five addition points is metered through five curved-tube flowmeters. The meters were calibrated for nitrogen flow by use of a wet-test meter as the primary flow standard. In order to obtain values for pounds per minute HF flow through the meters, it is necessary to know the pressure and temperature of the HF gas passing through the calibrated meters and the state of isomerization of HF as a function of pressure and temperature. From values found in the literature on the average molecular weight of HF gas as a function of temperature and partial pressure (Fig. 2), the mass feed rate to the reactor can be calculated, using the volumetric calibration.

The first work with the reactor will be its use as a countercurrent moving-bed drier for the UF_4 feed to the oxidation reactor.

Oxidation. In the rebuilt oxidation reactor (CF-56-6-100, Figs. 3 and 4) the cold trap, a 1-ton fluorine cylinder, has been relocated on top of cell 2. Steam-traced tubing connections have been made from the oxidation reactor through the cell top to the trap. On the vent line from the trap a connection is to be made to a hydrolyzer column for recovery of UF_6 , as UO_2F_2 , from the off-gas line. Two molar caustic will be recirculated from a polyethylene tank through a 3-in. inconel column packed with 1/4-in. nickel rings. The cold trap will be warmed, vented

Fig. 2. Effect of Temperature and Partial Pressure
on Apparent Molecular Weight of Gaseous HF
 [Chem. Revs. 8:222 (1931)]

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through the column, and purged with dry nitrogen to rid it of UF_6 . The caustic tank will be sampled for uranium to facilitate making a material balance on the oxidation system.

Graphite pellets, which will be mixed with UO_2F_2 pellets for a preliminary oxidation run, were prepared by grinding graphite column packing and scrap reactor-grade graphite rod stock. A combination of a jaw-crusher and the Stokes granulator was adequate for the preparation. The purpose of the preliminary run is to dry the U308 reactor lines and to test the temperature measurement and control system. UF_4 pellets for the first oxidation run will be dried in the reduction-hydrofluorination reactor.

3.0 EXCER PROCESS

The Excer process was originally designed to prepare UF_4 from $UO_2(NO_3)_2$ by ion-exchange conversion of the nitrate to uranyl fluoride and electrolytic reduction of this compound, followed by dehydration, to UF_4 . The process is being applied directly to unfiltered ore leach. In this case the uranium is first converted to uranyl chloride and then to uranyl fluoride, both by ion exchange, or the chloride is electrolyzed directly.

3.1 Laboratory-Scale Studies (I. R. Higgins)

Ion Exchange. In runs with contaminated synthetic ore leach in the 2-in.-dia continuous anion-exchange column, UO_2Cl_2 product containing as much as 225 g of uranium per liter was obtained. The product concentration increased as the runs continued, being only 78 g/liter in the first run. Product sodium concentrations were as low as 0.05 M.

In later runs, 8 M HCl was used to remove sulfate from the ion-exchange columns, and the uranium was then eluted with water. The loading section of the column was 3 ft high and contained 20-30 mesh Permutit SK resin. With a loading of 40 g of uranium per liter, recovery was greater than 99.8%. About 3 lb of HCl was consumed per pound of uranium. In process, the choice between NaCl and HCl would be made on the basis of such factors as cost, corrosion, and objection to the presence of sodium.

The boron concentration in the Excer UF_4 product has sometimes been 4 or 5 times as high as that in the feed. It appears that this excess boron is introduced from equipment or in one of the reagents. In experiments in which boron was added to the feed to 10 wt % of the uranium, it was removed by factors of 104 and > 660 in the anion- and cation-exchange steps, respectively. In the anion-exchange column it was weakly sorbed with about two-thirds breaking through in the

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feed waste and the other one-third removed in the 8 M HCl elution. In the cation-exchange step, the boron acted as an anion and was not sorbed. Cadmium added to the feed to 10 wt % of the uranium was decontaminated by a factor of >1000 in the ion exchange step. About half appeared in the feed waste and 8 M HCl eluate. During the water elution of the UO_2Cl_2 product, the other half of the cadmium remained on the resin. This indicates that the resin may have to be thoroughly cleaned occasionally to prevent cadmium buildup.

Reduction and Dehydration. The UO_2F_2 product was made 2 M in H_2SO_4 and reduced in the 6-by-6-in. electrolytic cell. The UF_4 hydrate was filtered on paper and dehydrated in a magnesium tube. About 1.5 lb of UF_4 was produced with a tap density of 3.1 g/cc. The specific surface area of the UF_4 was $1.1 \text{ m}^2/\text{g}$.

A series of electrolyses was made, using either HCl or H_2SO_4 in varying concentrations to carry the current in the catholyte, to observe the effect on product tap density. No consistent or remarkable differences were noted. Products from UO_2Cl_2 prepared by ion exchange were better than those from UO_2Cl_2 prepared from UO_3 :

Catholyte			Tap Density (g/cc)	
U (g/liter)	F ⁻ (M)	Acid	Before Grinding	After Grinding
Synthetic Feeds				
120	2	1 M HCl	2.3	2.5
120	2	0.5 M H_2SO_4	1.8	1.9
120	2	3 M HCl	2.0	2.3
120	2	3 M H_2SO_4	1.8	2.5
Ion Exchange Product Feeds				
119	2	2.1 M HCl	1.8	2.9
65	1.1	4.3 M HCl	1.9	3.0

The current density was 3 amp/in.² and the temperature was 91-95°C in these runs.

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The 6-in. laboratory electrolytic cell was later provided with a bottom product take-off in order to eliminate the agitation pump (Vanton). All products from this cell have been very granular, fast settling, and fast filtering but with very low tap densities, 1.5 to 1.8 g/cc. This extreme in particle size and tap density had not been encountered before. Particle size measurements indicated that large particles were only agglomerates of smaller particles. The tap density of the dehydrated product was increased from 1.5 to 3.0 g/cc by grinding in a mortar and pestle. However, the density of a product already on hand was increased only from 2.2 to 2.4 g/cc by grinding, and the density of another was increased only from 3.0 to 3.1 g/cc.

A sample of UO_2Cl_2 -6 M HCl strip product from the organic phosphate extraction system from the Bureau of Mines in Salt Lake City was electrolyzed without pretreatment. Analyses are not yet complete, but the amounts of potassium, aluminum, and rare earths in the product indicate that an ion-exchange cleanup of this material before electrolysis is necessary.

3.2 Larger-Scale Studies (C. W. Hancher)

Reduction. Approximately 10 lb of $\text{UF}_4 \cdot 3/4\text{H}_2\text{O}$ has been continuously produced by electrolytic reduction of uranyl fluoride in an ion-exchange membrane cell. The tap density of the $\text{UF}_4 \cdot 3/4\text{H}_2\text{O}$ product is 2.6 g/cc. The conditions of the run were:

Feed composition (catholyte)	0.25 M UO_2^{++} , 0.5 M SO_4^{--} , 1 M + 10% excess HF
Feed rate	19.0 liters/hr (3.8 moles U per hour)
Temperature	95°C
Current	300 amp at 12.4 volts
Current efficiency	52%
Reduction efficiency	76%
Anolyte, in	0.5 M H_2SO_4
out	0.5 M H_2SO_4 , 0.1 g U and 0.19 g F per liter

The uranium was reduced at a rate of about 0.018 mole/amp-hr. The product contains 73.9% U and 22.6% F.

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Fig. 3. 3-in.-dia Nickel Fibron Flite Dehydrator

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Fig. 4. 36-in.-dia Resin Test Loop.

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FIG. 5

SCHEMATIC DIAGRAM OF A HYDRAULIC ACCUMULATOR
TO REPLACE THE RESIN PULSE PUMP FOR
A HIGGINS ION EXCHANGE CONTACTOR

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Dehydration. Installation of the 3-in.-dia 6-ft-long nickel ribbon flite dehydrator (Fig. 3) was completed. In a test run, $UF_4 \cdot 3/4 H_2O$ (3.48% H_2O) was fed at a rate of 3 lb/hr. The product UF_4 contained 1.47% H_2O , which is about three times the specification value. The temperature in the contactor was

First foot	120°C
Next 2 ft	250°C
Last 3 ft	380°C

The hold-up time in the dehydrator was about 35 min.

4.0 ION-EXCHANGE ORE PROCESSING (C. W. Hancher)

Intermittent operation of a resin pulse pump with a large-scale --36 in. dia or larger--contactor (Fig. 4) is cumbersome. Engineering design of a hydraulic accumulator (Fig. 5) has therefore been started to replace the 25-hp 200-gpm pulse pump that would be required for this large contactor.

MWG:mep

Previous reports in this series were CF 56-5-100, for May (30%), and CF 56-6-100 (30%), for June. The next CF 56-8-100 (30%) will be published shortly.

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