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For The Atomic Energy Commission

H. F. Canale

Chief, Declassification Branch

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CHAMBERS WORKS PROCESS
FOR THE MANUFACTURE OF
PERURANIC ACID

By

G. M. Richardson

OPTICAL
CENTER

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BP-13

APPROVED: NOVEMBER 15, 1944

CHAMBERS WORKS PROCESS
FOR THE MANUFACTURE OF
PERURANIC ACID

(330.20)

HISTORICAL

This process was developed in Jackson Laboratory (Progress Report JAD-13) and was further developed in the Blue Products Area plant. It supersedes this section of the second revision of the "Tentative Chambers Works Process for the Manufacture of C-112", dated August 19, 1944.

PATENT STATUS

No investigation of the Patent Status has been made by the Operating Department.

PRINCIPLES

Uranyl sulfate solution is clarified by filtration and precipitated as peruranic acid by means of hydrogen peroxide under controlled pH conditions. The precipitated peruranic acid is isolated in a filter press or centrifuge. The following equation indicates the reaction which occurs -



YIELD

The yield in this operation is about 99.0% theoretical. About 1,911-lbs. of Uranium are obtained per charge as a paste of Peruranic acid.

EQUIPMENT (See Attached Flow Sheet)

EP 228 Cooling Tank (F 60592)

A 12' x 10' steel tank, lined with neoprene. Equipped with agitation, stainless steel steam sparger and Carbate cooling coils. 588-lbs. per inch (water).

EP 233 Polishing Press (BPP 68972)

A 24" wooden press with 10.5 cu. ft. capacity.

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EP 220 Precipitation Tank (D 60584)

A 12' x 10' steel tank lined with neoprene and equipped with a large agitator and a small "Lightain" agitator. A Northite Pump is also part of the agitation system for this tank since it is used to circulate and mix the peruranic acid slurry during caustic addition. The tank is heated with a stainless steel steam sparger and cooled with Farbate coils using river water. 588-lbs. per inch (water).

EP 230 Hydrogen Peroxide Measuring Tank (D 60593)

A 6' x 5' stainless steel tank. 161-lbs. per inch (hydrogen peroxide).

EP 231EP 232 Caustic Measuring Tanks (D 60594)

Two 8' x 5' steel tanks. 339-lbs. per inch (caustic).

EP 222 Continuous Centrifuge (BPF 68955)

A 24" stainless steel, solid bowl continuous centrifuge. Peruranic acid paste is delivered to a portable, agitated stainless steel slurry buggy or to barrels. The effluent goes to the batch centrifuge.

EP 223 Batch Centrifuge (BPF 68996)

A 40" stainless steel, solid bowl Bird centrifuge. Cake is ploughed batchwise to drums while the effluent delivers continuously to EP 224 or the settling tank.

EP 224 Effluent Tank (D 60588)

A 6' x 5' stainless steel tank. 147-lbs. per inch (water).

Settling Tank (DW 5158)

A 20' x 9' 1/4" wooden tank, equipped with very slow agitation. Cake discharges from a cone in the bottom to 203 tanks by means of a pump. 1,360-lbs. per inch (water).

EP 226EP 227 Peruranic Acid Slurry Tanks (D 60590)

Two, 10' x 10' stainless steel tanks, equipped with agitation and steam spargers. 409-lbs. per inch (water).

Peruranic Acid Isolation Presses (DW 5181)

Two, 36" rubber filter presses with a combined capacity of 75 cu. ft.

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Sump

A 100 cu. ft. concrete catch basin for floor drainage delivering to the settling tank by means of a steam siphon.

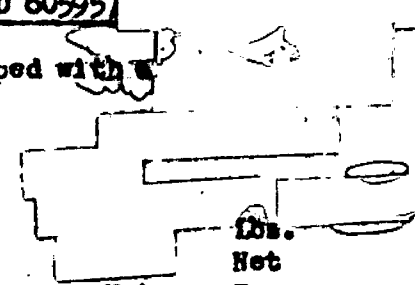
EP 234 Barnstead Still (BPP 68966)

Capacity, 300-gals. per hour distilled water.

EP 235 Distilled Water Storage Tank (D 60595)

A 10' x 10' stainless steel tank equipped with a steam coil for heating. 407-lbs. per inch.

PROCEDURE



<u>Materials</u>	<u>Mol. Wt.</u>	<u>Lbs. Net</u>	<u>Lbs. 100%</u>	<u>Mols. Per Mol.</u>	<u>Net</u>	<u>Lbs. Net</u>
					<u>Parts U Used</u>	<u>Per 100-lbs. U Produced</u>
Uranyl Sulfate Solution	366.28	52920	1930*	1.000	27.4	2768
Hydrogen Peroxide (27.5%)	34.02	1640	451	1.635	.85	85.8
Soda, Caustic Liquid (30%)	40.01	2700	810	2.495	1.4	141.3
Glue, Fish	--	100	5	--	.05	5.2
Peruranic Acid	338.20	4270	1911*	.990	2.21	223.2

* As uranium. All values refer to uranium (Mol. Wt. = 238.14) in this process and not to uranyl sulfate or peruranic acid.

The uranyl sulfate solution is filtered through the press to the precipitation tank where the charge is heated to 38°C. - 42°C. with live steam. The peroxide is added over a period of 10 minutes to 15 minutes and the charge is allowed to agitate for approximately 30 minutes until the pH reaches a minimum (1.1 to 1.9).

Hazards

Wear rubber gloves and goggles when handling peroxide because it causes severe burns.

Hydrogen peroxide causes combustion when exposed to wood. For this reason any spills on the carboy frames should be washed freely with water. Hydrogen peroxide should not be stored on wooden floors.

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Peruranio Acid

When this point is reached circulation of the charge through the pump is started and the caustic soda solution is added to the circulating liquor on the suction side of the pump at such a rate that the pump discharge at the tank is strongly alkaline to Brilliant Yellow and faintly alkaline to phenolphthalein (8.0 to 8.5). While circulating, the pH of the main part of the charge is held below 3.4 and the temperature is kept between 40 C. - 45 C. As the pH of the charge approaches 3.0, caustic addition is made more cautiously until the pH of the charge reaches 3.2 ± 0.2 .

Final Conditions

Temperature	40° C. - 45° C.
pH	3.2 ± 0.2
Volume (58,800-lbs. to 64,680-lbs.)	100" to 110"
Starch - KI Spot	Definite excess of peroxide.
Congo red spot	Brown
Potassium Ferrocyanide	No soluble uranium
Appearance	Creamy slurry, colorless solution.

When the final conditions of the charge are adjusted as indicated above, the glue solution is added and the charge is centrifuged, the cake going to EP 226 and the effluent going to the settling tank.

The volume in EP 226 is finally adjusted to 90" with filtered water. The charge is heated to 40°C. - 45°C. if the pH is below 4.5 and 60°C. - 65°C. if the pH is above 4.5. The charge is again centrifuged with the cake going to EP 227 and the effluent to the settling tank. The volume in EP 227 is adjusted at 90" with distilled water and the charge is heated to 60°C. - 65°C. and centrifuged. In this case the cake (peruranic acid paste) is discharged to drums with the effluent going to the settling tank.

Alternate Method of Isolation

The charge in the precipitation tank may be pumped to the presses and washed sulfate free with the filtrate going to the settling tank. This method is used on charges which do not centrifuge easily.

Settling Tank

All effluents and filtrates deliver to the settling tank where the solid matter containing uranium is settled and pumped to the dissolving, oxidizing and liming tanks. The liquor flows over a wier to the ditch.

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Peruranic Acid

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Plant Capacity

The capacity of this operation depends on the uranium content of G & R Feed produced in another operation. The capacity of the U & R operation is 250 tons per month of raw feed of variable composition. Current feed stocks are yielding approximately 70,000-lbs. uranium as peruranic acid per month.

Specifications

Hydrogen Peroxide

Approximately 27.5% H₂O₂

Soda Caustic

Approximately 30% NaOH,
Low NaCl and Boron content.
Specification - No. 73-S

Peruranic Acid

B.P. Laboratory Method
No.

Boron	< 2.5 p.p.m.	112.46
Fluorine	< 0.25% Average, 0.5% max.	112.50
Sulfates	< 1.0%	112.44
Purity (% Uranium)	> 68.0% (Dry basis)	112.42
Solids	No Specification	112.41

Hazards

Caution should be exercised to avoid spillage or contact with the uranyl sulfate solution or the peruranic acid slurry. If any material should be spilled or splashed on the skin or clothing it should be washed off with water.

A daily change of clothing and a shower are required. All personnel must be examined by the Medical Department every three weeks.

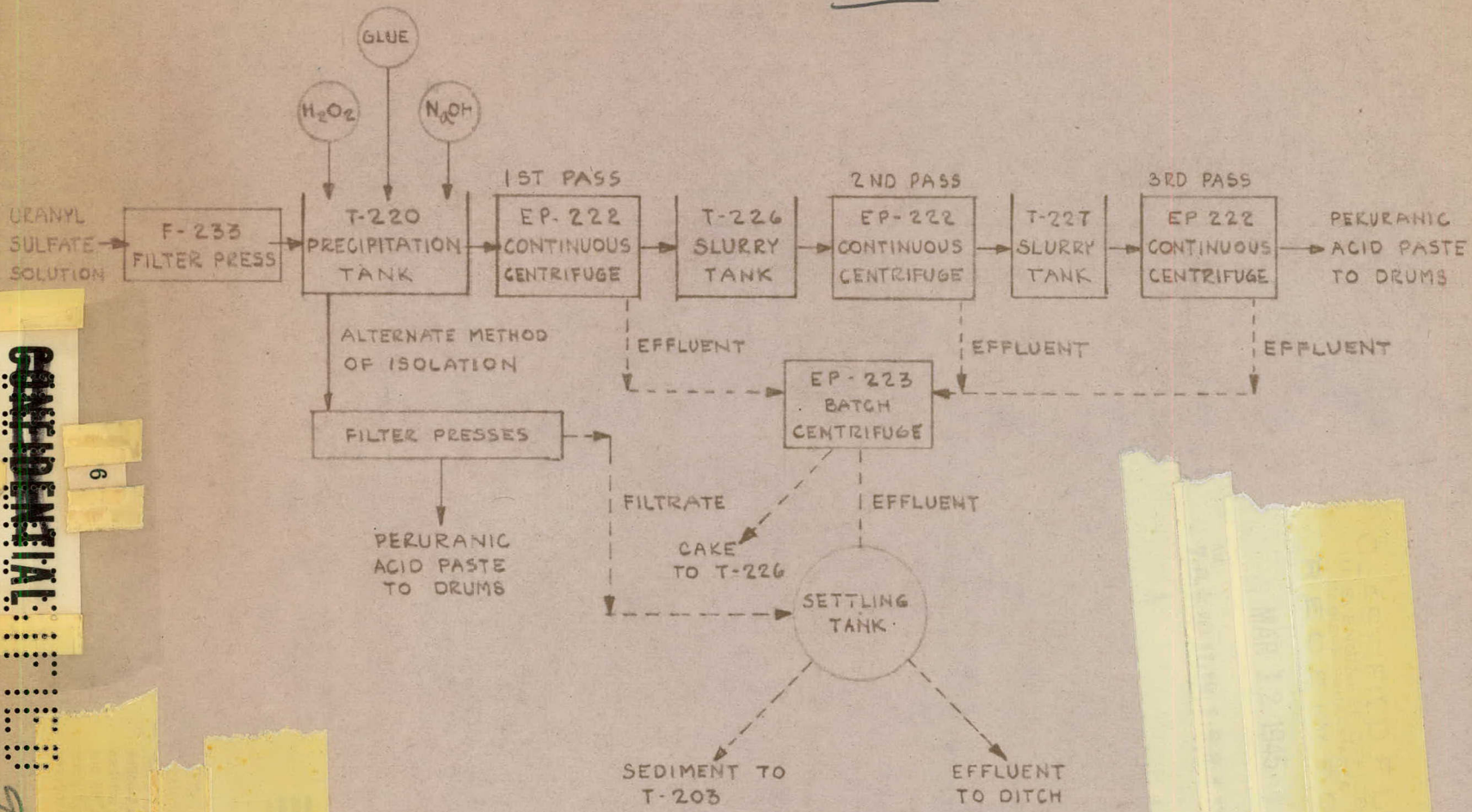
Goggles and rubber gloves must be worn when handling caustic or hydrogen peroxide. Hydrogen peroxide causes combustion when allowed to come in contact with wood. For this reason carboys should not be stored on wood floors. When peroxide comes in contact with wood it should be washed immediately with water.

G.M. RICHARDSON,
BLUE PRODUCTS AREA.
NOVEMBER 15, 1944

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NOTE:
 EP-222 USED FOR ALL 3 PASSES
 AS SHOWN ABOVE.

PROCESS FOR MANUFACTURE OF
 PERURANIC ACID SOLUTION
 FLOW SHEET

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