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ELECTRODEPOSITION RESEARCH PROGRESS REPORT

[redacted]

[redacted]

Note: Effective March 1, 1948 this report will be issued the first of every month instead of bi-monthly.

Date: March 1-31, 1948

[redacted]

Prepared by: Edward Orban

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ELECTRODEPOSITION RESEARCH GROUP

W. Abel, R. Bell, E. Orban, and W. Raiff

ABSTRACT

Plating of Postum out of Nitric Acid and Hydrofluoric Acid Solutions - W. Abel and W. Raiff

Postum was plated out of 1.5 normal nitric acid and 1.0 normal hydrofluoric acid under similar conditions. The hydrofluoric acid plate was better appearing, and had a much higher curie density. However, neutron counts in these runs were quite similar.

Neutron Counts - W. Abel and W. Raiff

A study of the change of neutron counts over a period of time was started.

Conversion of Active Hydrofluoric Acid Solutions to Hydrochloric Acid Solutions - W. Raiff

A 99.99^{+} per cent conversion was effected.

Conversion of Production Solutions to 1.0 Normal Hydrofluoric Acid Solutions - R. Bell

Good conversion has been achieved; however, further work needs to be done on complete change of postum from the production solution to 1.0 normal hydrofluoric acid solutions.

DETAILED REPORT

Plating of Postum out of Nitric Acid and Hydrofluoric Acid Solutions - W. Abel and W. Raiff

In order to make a comparison, two plating runs of postum on platinum were made; one out of hydrofluoric acid, and one out of nitric acid. A tabular compilation of the results is shown in Tables I and II.

The foils plated from nitric and hydrofluoric acids were rinsed for three minutes in 1.5 normal nitric acid with 50 ma. current flowing between cathode and anode. The voltage was maintained at 3^{+} volts. The water rinse was carried out for two minutes with 3 volts between the electrodes. The current ranged from 0.3 to 0.7 ma. The final rinse in alcohol lasted two minutes.

The following points of comparison may be made:

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1. Per cent plated out of nitric acid compared favorably with the per cent plated out of hydrofluoric acid under the same conditions.

2. A higher line voltage is required and a higher amperage is observed in plating nitric acid solutions.

Table I

POSTUM PLATED ON PLATINUM FROM 1.5 NORMAL NITRIC ACID

Foil No.	533	534	535	536	537	538
Cathode Potential vs. N.C.E. (v.)	0.0	0.0	0.0	0.0	0.0	0.0
Ave. Current Density (ma./cm.^{2})	2.0	2.0	Poor electrical connection	2.4	2.4	2.4
Applied voltage Ave. (v.)	1.4	1.3		1.5	1.3	1.3
Plating Time (min.)	316	323		332	338	345
Calorimetric Assay (c.)	0.552	0.535		0.698	0.681	0.572
Case density (c./cm.^{2})	2.216	2.152		2.808	2.744	2.296
Neutrons/sec./c.	112	61		63	55	0

Table II

POSTUM PLATED ON PLATINUM FROM 1 NORMAL HYDROFLUORIC ACID

Foil No.	539	540	541	542	543	544
Cathode Potential vs. N.C.E. (v.)	0.0	0.0	0.0	0.0	0.0	0.0
Ave. Current Density (ma./cm.^{2})	0.4	0.8	0.75	0.24	1.2	2.0
Ave. Applied Voltage (v.)	0.68	0.62	0.43	0.56	0.66	0.57
Plating Time (min.)	348	352	359	365	371	378
Colorimetric Assay (c.)	0.511	0.502	2.064	0.663	3.896	4.415
Case Density (c./cm.^{2})	2.044	2.008	8.204	2.520	15.520	17.560
Neutrons/sec./c.	96	96	23	43	27	26

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3. The appearance of the postum plated from nitric acid was dark gray to black; whereas, that plated from hydrofluoric acid was light gray with a sheen on the surface.

4. It was expected that the nitric acid plated material would have a higher neutron count; however, since the counts from the two solutions is essentially the same it may be that the neutron count is a function of the manner in which the material is handled.

5. Foil No. 544 is of particular interest because of its high curie density. On the basis of 4.405 sq. cm. per production gauze, it would be possible to plate about 77 curies on the standard production gauze.

6. As large amounts of polonium were plated out the neutron count falls off. This, and the fact that admission of oxygen to the container increases the neutron count, indicates that the phenomenon may be a surface one.

Neutron Counts - W. Abel and W. Raiff

It has been of interest to follow the neutron counts of the foils over a period of time. Checks have been made on the foils shown in Tables III and IV.

Table III

CHANGE OF NEUTRON COUNTS WITH TIME

Foil No.	n./sec./c. March 10	n./sec./c. March 12
539	96	70
540	96	94
541	23	19
542	43	0
543	27	22
544	26	32
	March 3	March 8
533	62	49
534	33	35
535	-	-
536	44	12
537	38	38
538	0	50

The results over a short period of time are not very conclusive, so neutron counts have been made as shown in Table IV.

Table IV

Date	Total Neutrons			
	529	530	531	532
1/30/48	0	0	0	0
2/12/48	0	12	24	24
2/20/48	0	0	112	0
3/15/48	38	21	138	0

Apparently low neutron counts are difficult to reproduce, but a continual increase such as Foil Number 531, is significant. A small amount of oxygen probably is leaking into the apparatus, giving increasing counts.

Conversion of Active Hydrofluoric Acid Solutions to Hydrochloric Acid Solutions - W. Raiff

A series of runs were made reconverting the hydrofluoric acid solutions into hydrochloric acid. The process has been used with success in the past by the Process Research Group - (Seminar Paper, April 1, 1946 by Carl Rollinson). The runs were combined and the resulting solution analyzed in two parts. The ammonium fluoride residue was also checked, in two parts, to determine the amount remaining. The recovery was better than 99.99 per cent. Table V records the data.

Table V

RECOVERY OF ACTIVITY FROM HYDROFLUORIC ACID

Solution	Volume	Total Activity
Hydrochloric Acid #1	950 ml.	3.29 c.
Hydrochloric Acid #2	950 ml.	11.20 c.
Ammonium Fluoride #1	2500 ml.	9.9×10^{-4} c.
Ammonium Fluoride #2	2000 ml.	4.5×10^{-4} c.

Conversion of Production Solutions to Hydrofluoric Acid Solutions - R. Bell

To determine the best conditions for the preparation of hydrofluoric acid solutions from production solutions, the following experiment was carried out:

1. Excess saturated sodium carbonate solution was added to 25 ml. of production solution containing 5.24 c.

2. The precipitate was filtered through a fine sintered glass filter stick and washed twice. At this point postum and bismuth carbonates were crystalline materials in the bottom of the vessel. The filtrate and wash waters were acidified and were shown by count to contained 0.675 c. of postum.

3. Forty-eight per cent hydrofluoric acid was added to the precipitate. The bismuth carbonate and postum carbonate reacted to form the corresponding fluorides. Postum fluoride went into solution; whereas, most of the bismuth fluoride remained on this filter. This method, then gives an addition removal of bismuth before plating.

4. The resulting solution was filtered through a "Teflon" filter disc. This filtrate contained 3.00 c. of postum.

5. The bismuth precipitate remaining on the "Teflon" filter disc contained 1.40 c. of postum as well. Collected data are shown in Table VI.

Since much postum remained in the precipitate and the first filtrate, the procedure was modified in the next run. In step 1, saturated ammonium carbonate was used in place of saturated sodium carbonate. In step three, hot 48 per cent hydrofluoric acid was used and the solution was stirred until cool. The results were somewhat better. (See Table VI).

In the third run the production solution was made alkaline with an excess of saturated sodium carbonate. The first filtration was made with a coarse glass filter stick, and the leaching of postum out of the carbonate precipitate was made in the following way: Ten ml. of warm (60°C.) 10 N hydrofluoric acid was added to the precipitate and maintained at 60°C. (using a platinum immersion heater) for 45 minutes with vigorous stirring. It was then allowed to cool to room temperature with continuous stirring.

It was found that less postum appeared in the first filtrate. (See Table VI).

The third run was repeated except that a fine glass filter was used to draw off the sodium carbonate solution. In order to determine where the postum was being lost, the apparatus was extracted with 200 ml. of concentrated hydrochloric acid. These results are also shown in Table VI. It was possible to remove 99.96 per cent of the postum from the production solution.

The postum remaining in the precipitate, and the postum unaccounted for suggests the course of immediate research.

Some of the equipment designed and constructed for this work is shown in Figures 1 through 6.

FUTURE PLANS

1. Work is under way on determining the relative cohesive qualities of postum plated on platinum out of 1.5 N nitric acid solution to that plated out of 1.0 N hydrofluoric acid solution.

2. Continued attempts to improve the per cent conversion of postum in production solutions to hydrofluoric acid solutions will be made.

3. Attempts to produce production size foils plated out of hydrofluoric acid will be made.

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480097 Figure 1

"Teflon" Filter Arrangement (Disassembled)

480096 Figure 2

Platinum Immersion Heater

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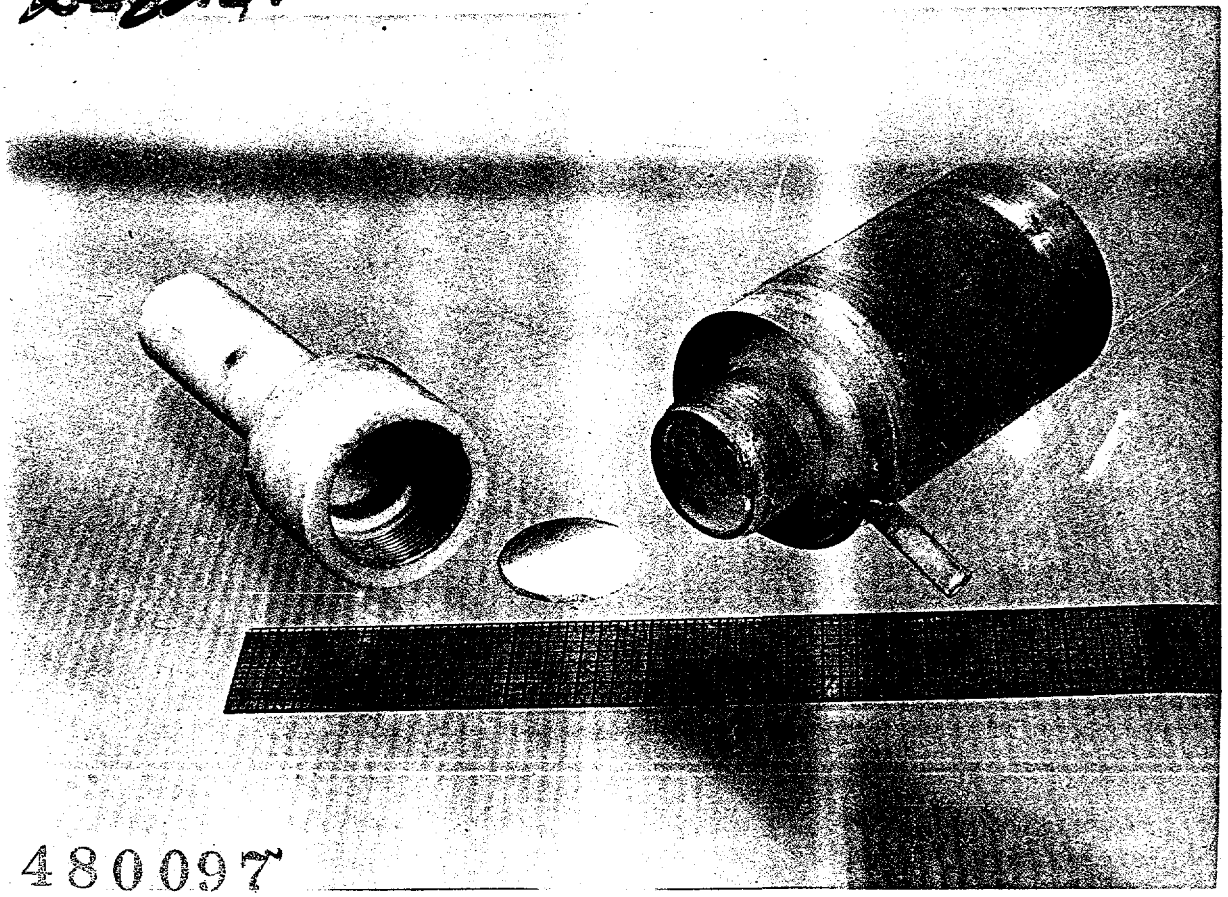

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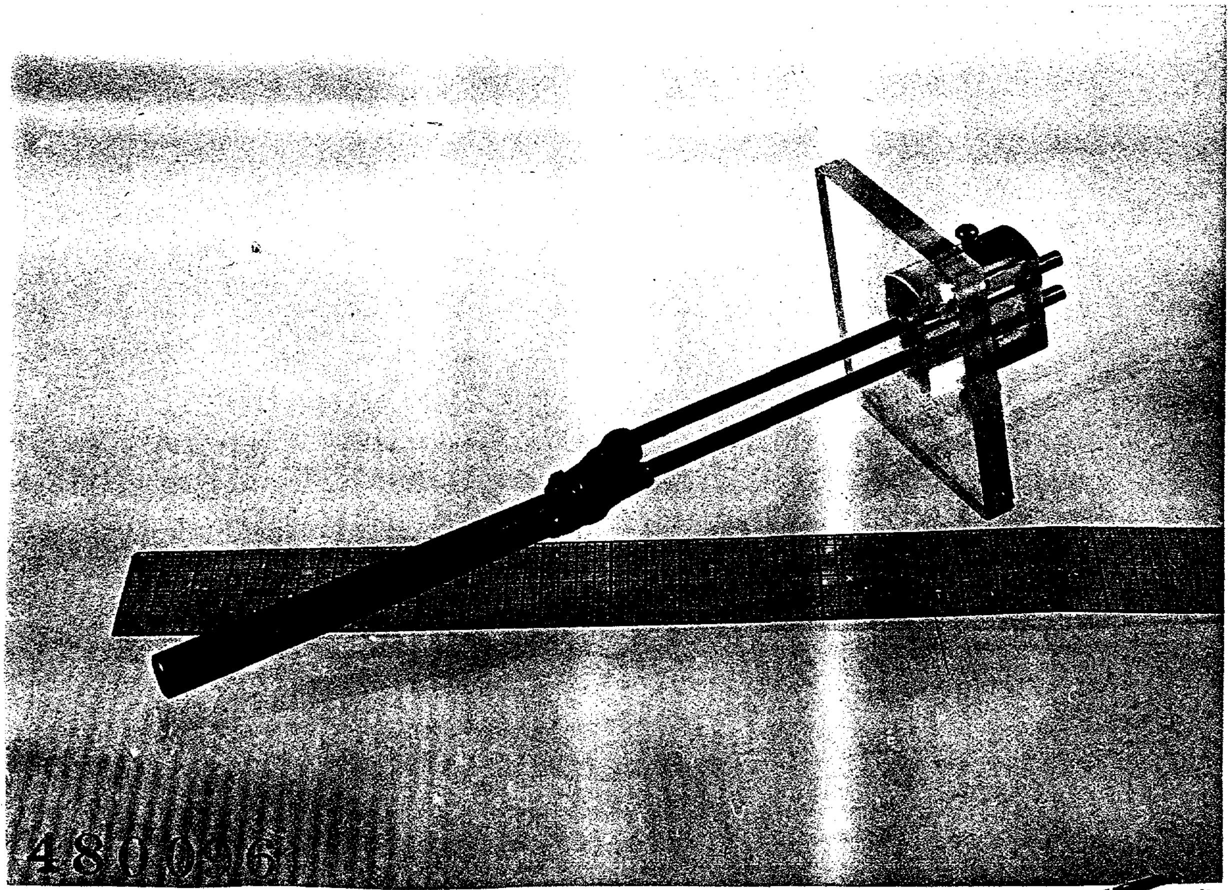

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480100 Figure 3

Polystyrene Reaction Flask for the
Neutralization of Production Solutions
with Sodium Carbonate

480101 Figure 4

Polystyrene Reaction Chamber with
Immersion Heater for Leaching of
Bismuth Fluoride Precipitate

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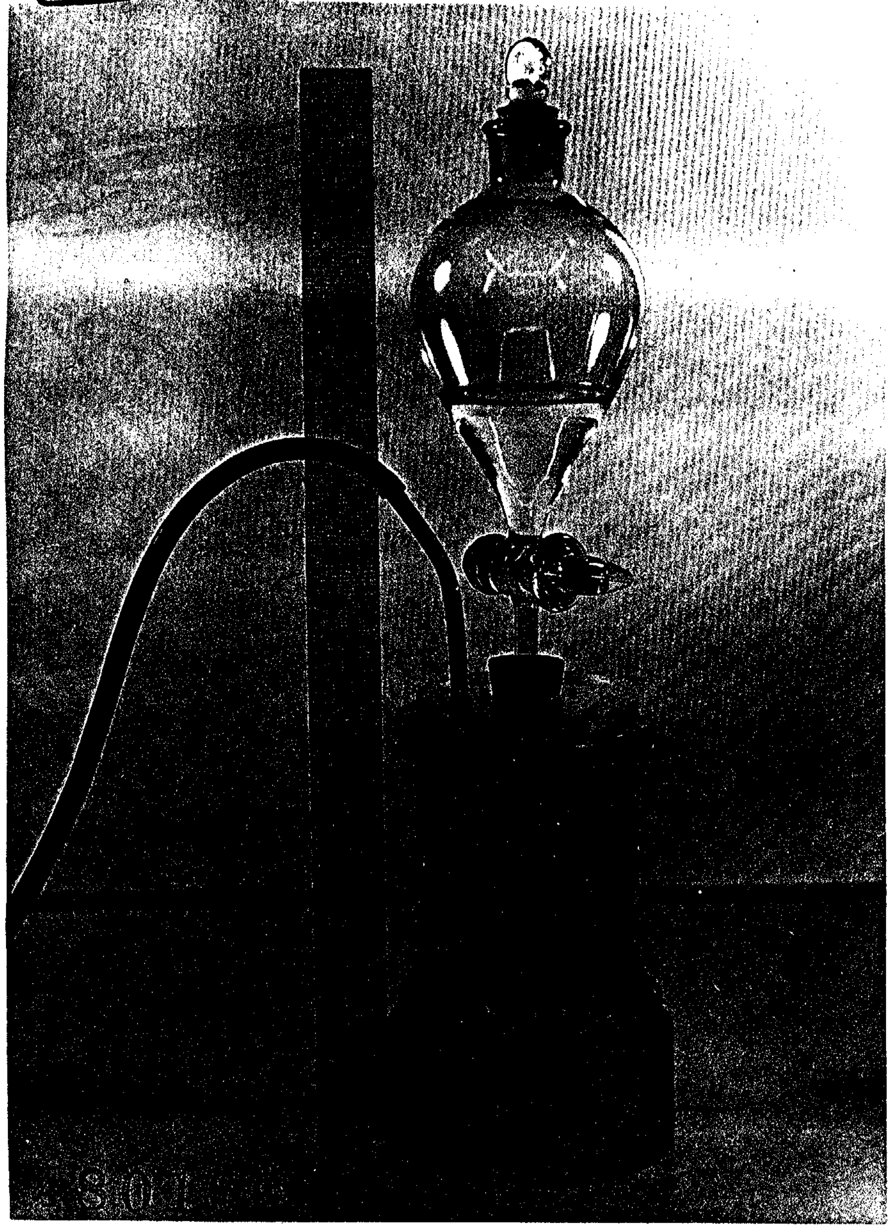

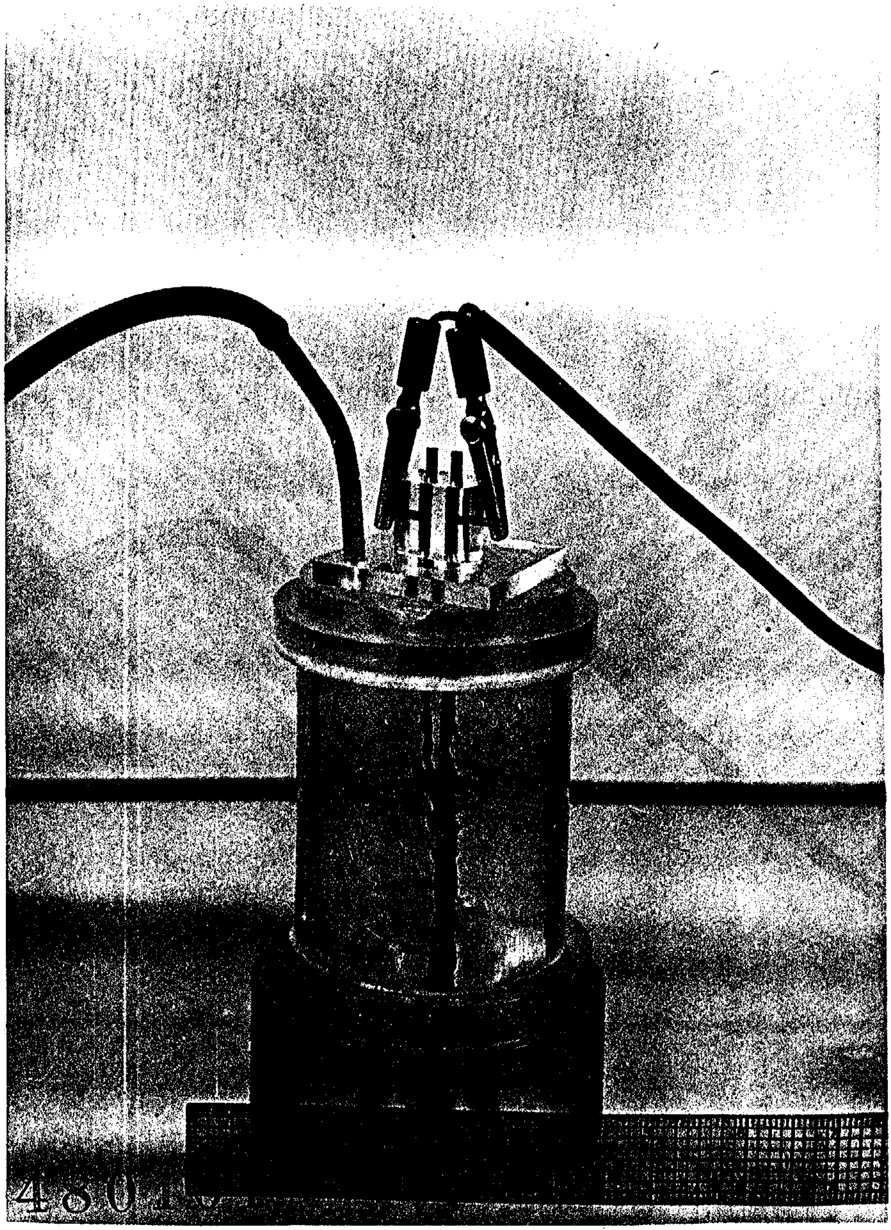

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Figure 5

Setup for Filtering Bismuth Carbonate and Postum Carbonate Precipitate using a Sintered Glass Filter Stick

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Figure 6

"Teflon" Filter Funnel and Polystyrene Vacuum Flask used in the Final Filtration of a Solution of Postum Fluoride from Bismuth Fluoride Precipitate

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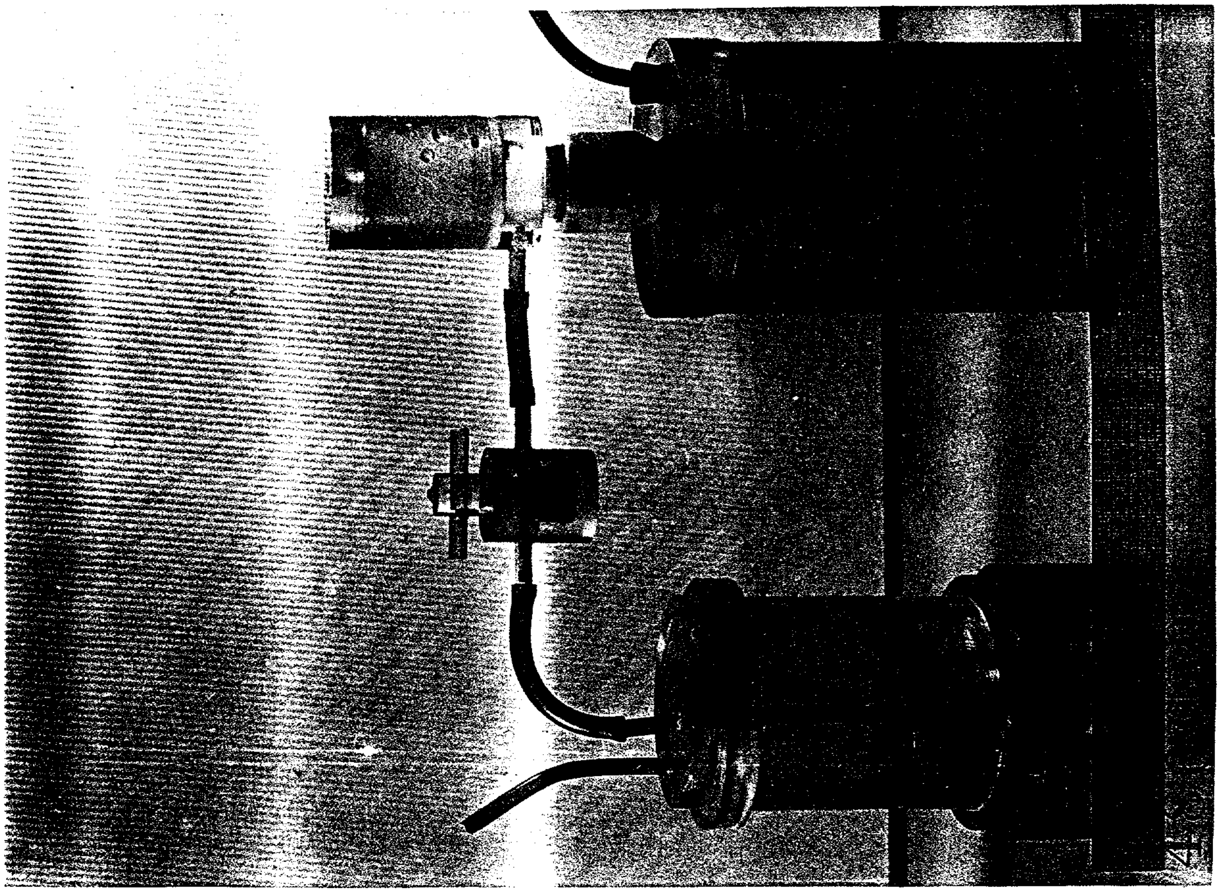

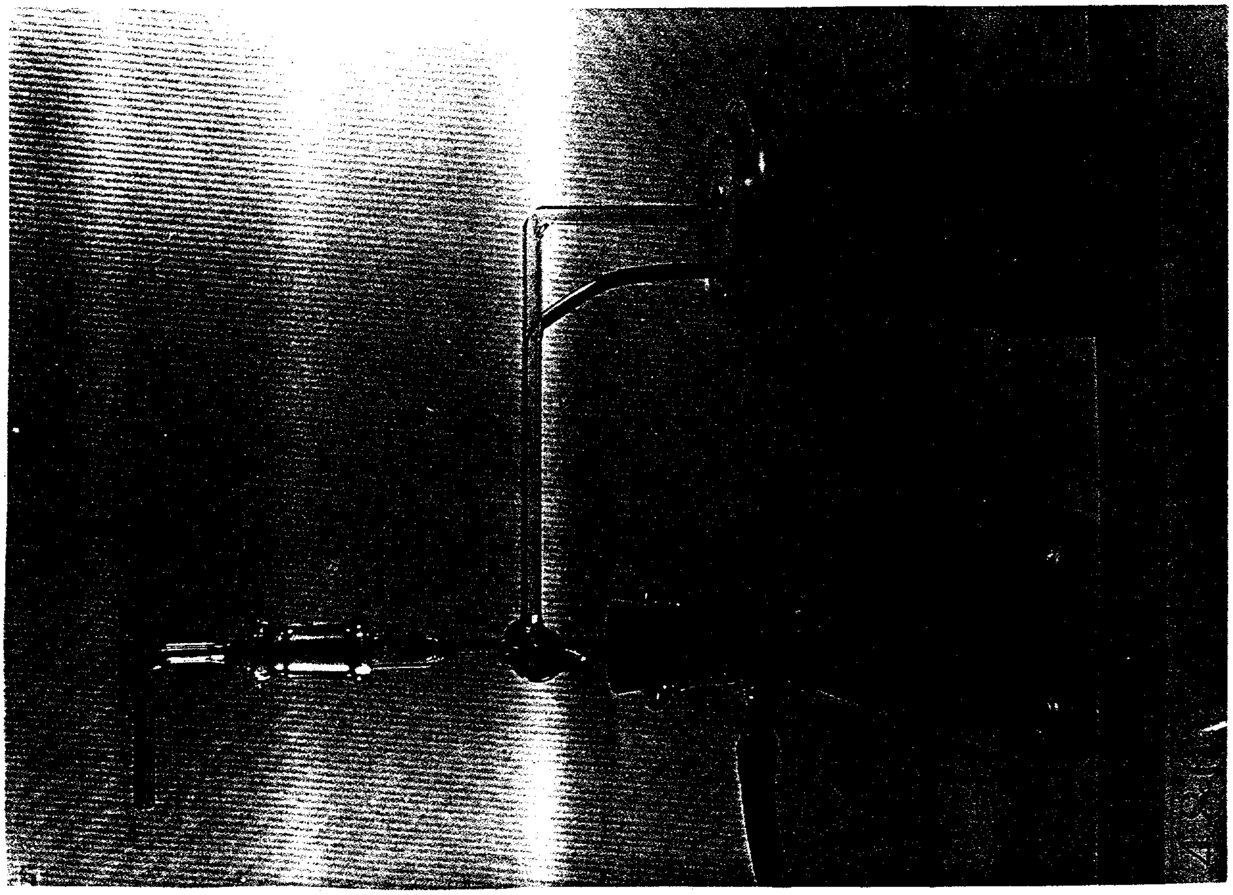

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Table VI

PREPARATION OF POSTUM IN 1.0 NORMAL HYDROFLUROIC ACID

Run No.	Total Postum in Original Solution	Postum Remaining in First Filtrate	Postum in HF Solution	Postum Remaining in Precipitate	Postum Removed from System with Hydrochloric Acid	Unaccounted for
1	5.24 c.	0.675 c.	3.00 c.	1.40 c.		0.16
2	5.22 c.	0.52 c.	4.05 c.	0.35 c.		0.30
3	4.95 c.	0.20 c.	3.78 c.	0.39 c.		0.58
4	4.90 c.	0.02 c.	3.70 c.	0.21 c.	0.16 c.	0.81

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