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MONSANTO CHEMICAL COMPANY - UNIT III

MLM-26

PROGRESS REPORT

MLM-MH-48-61-0021

January 16-31, 1948

ELECTRODEPOSITION RESEARCH GROUP

Abel, Bell, Orban, and Raiff

MLM-26

ABSTRACT

Solubilities - Orban

The solubility of postum in various concentrations of nitric acid was determined at 25.4°C.

Hydrofluoric and Trifluoroacetic Acids - Abel and Raiff

Details of this work will be reported in the next Progress Report.

Conversion of Nitric Acid Solutions to Hydrofluoric Acid Solutions - Bell

Conversion of production solutions to hydrofluoric acid solutions was tried by precipitation with ammonium oxalate, aluminum hydroxide, ammonium hydroxide, and sodium carbonate. Silver and Teflon discs were used.

DETAILED REPORT

Solubilities

The solubility measurements at 25.4°C. were made in a way similar to that described in the Progress Report of October 1-15, 1947.

Measurements of the heating effect of the activity in solutions were made. Table I shows the data.

Table I
Heating Effect of Postum

Solution No.	Normality HNO_3	Bath Temperature	Solution Temperature
8	0.1036	25.4° ± 0.03°C.	26.1°C
9	0.5046	"	26.1°
11	1.006	"	26.1°
6	1.595	"	26.1°
7	2.081	"	26.9°

DISCLAIMER

The concentration of postum in acid solutions is shown in Table II.

Table II

Solubility of Postum in Nitric Acid at 25.4°C.

Age of Solution (days)	Solubility in Units/ml. Acid Solution				
	0.1036N	0.5046N	1.006N	1.595N	2.081N
1	0.044	0.096	0.177	0.333	0.609
2	0.018	0.067	-	0.365	0.536
3	0.019	0.071	0.269	0.477	0.824
4	0.020	0.070	0.131	0.409	0.599
5	-	-	-	0.368	0.538
6	0.018	0.062	0.128	0.414	0.452
7	0.063	0.123	0.132	0.218	0.283
8	-	-	0.132	0.155	0.238
9	0.086	0.152	0.130	0.134	0.205
10	0.018	0.062	-	-	-
11	-	-	-	0.116	0.168
12	0.020	0.065	-	0.205	0.420
13	0.018	0.065	-	-	-
14	0.020	0.067	-	0.208	0.244
15	0.020	0.082	-	0.105	0.172
16	-	-	0.127	-	-
17	-	-	0.134	0.107	0.173
18	-	-	-	0.105	0.172
19	-	-	-	0.107	0.176
22	0.019	0.073	-	-	-
23	0.018	0.075	-	-	-
27	-	-	-	0.109	0.168

These data are shown graphically in Figure 1. As has been noted before, the change in solubility is very marked in the higher concentrations, and becomes less in the more dilute acid solutions. In the case of 1.595 normal and 2.081 normal nitric acid solution the change is so rapid that it is difficult to extrapolate to zero time. Before any conclusions are drawn it will be necessary to recheck the solutions. The solubilities, as obtained from the extrapolation, are shown in Table III.

Table III

Solubility of Postum

Normality of Nitric Acid	Solubility of Q (Units/ml.)	Solution Temperature
0.1036	0.019	26.1°C.
0.5046	0.072	26.1°C.
1.006	0.131	26.1°C.
1.595	?	26.1°C.
2.081	0.628 (?)	26.9°C.

Conversion of Nitric Acid Solutions to Hydrofluoric Acid Solutions - Bell

Continuing the attempt to find a satisfactory method of precipitating and filtering postum and bismuth from production solutions, in order to convert to hydrofluoric acid solution, the following experiments were run:

(1) Using a sintered silver filter (1/2 inch in diameter) 10 ml. of solution containing 0.0896 units was filtered after precipitation with ammonium oxalate. The filtrate was collected and assayed. The precipitate was dissolved in 1.5 N nitric acid and also assayed. Fifty per cent of the activity could not be accounted for, but is was found to have remained undissolved on the filter.

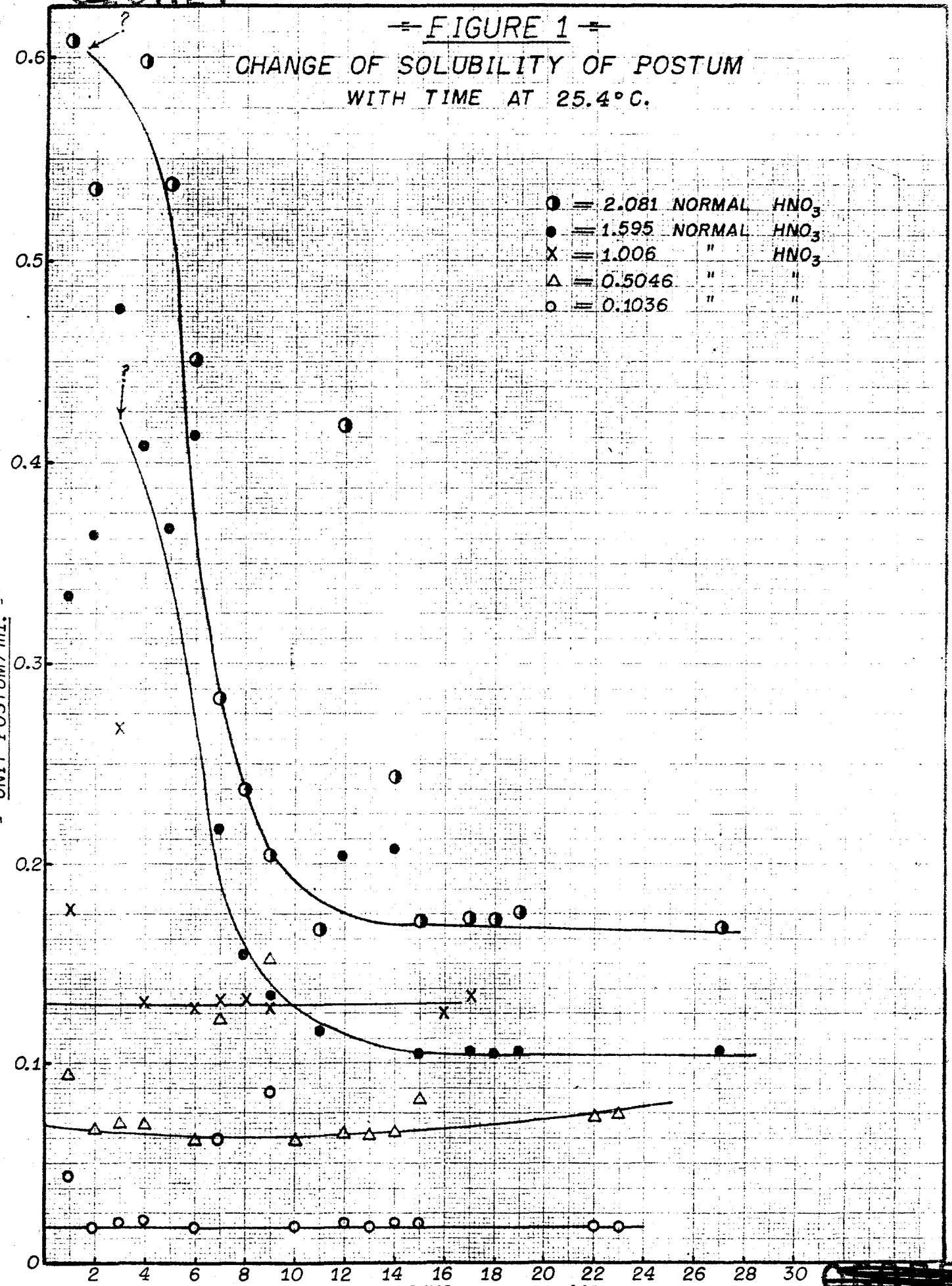
(2) Using the same silver disc, 0.092 units per 10 ml. was filtered through after precipitation in the presence of aluminum hydroxide using ammonium hydroxide (pH was adjusted to a value of 9). Most of the activity remained in solution.

(3) 0.0896 units/10 ml. was precipitated with sodium carbonate at a pH of 5. It was filtered twice through an 1/8 in. thick sheet of porous Teflon gasket material. Only a small percentage filtered through.

(4) A run similar to the previous one was made except that only one filtration was made.

FIGURE 1

CHANGE OF SOLUBILITY OF POSTUM WITH TIME AT 25.4° C.



Electrodeposition Progress Report

The results are complied in Table IV.

Table IV

Precipitation of Postum in Various Media and Filtration Through Silver and Teflon Discs

Run	Filter Disc	Total Activity	Precipitant	No. of Passes	% of Activity	
					in filtrate	in ppt.
1	Silver	0.0896	$(NH_4)_2C_2O_4$	1	6	94
2	Silver	0.092	$Al(OH)_3$ NH_4OH	1	99.5	0.4
3	Teflon	0.0896	Na_2CO_3	2	0.6	99.2
4	Teflon	0.0896	"	1	0.7	96

An apparatus is being constructed which will permit production solutions to be precipitated, filtered, and the precipitate dissolved in hydrofluoric acid, all in a closed system. Porous Teflon will be used as the filter medium, and the production solutions will be precipitated with sodium carbonate solution.

Hydrofluoric Acid and Trifluoroacetic Acid - Abel and Raiff

Work on these acids will be reported on in the next Progress Report.

FUTURE PLANS

Details will appear in the next Progress Report.

Edward Orban

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