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MONSANTO CHEMICAL COMPANY - UNIT III

PROGRESS REPORT

January 16-31, 1948

PRODUCTION RESEARCH GROUP

Blanke*, Hazlett, Kirby*, Nelson, Payne, Rogers, and Stringham

* temporarily transferred to Y-group.

ABSTRACT

I. The equipment for the Bismuth-II process has been mounted and is ready for installation at Unit IV.

II. The major portion of the gamma activity in the aluminum jackets is known to consist of iron and one of the following elements: zinc, cobalt, manganese, and nickel. Accordingly, a separation of these elements from aluminum should be obtained by precipitation from a concentrated sodium hydroxide solution to which sodium peroxide is added. Experiment showed that 88 per cent of the gamma activity was separated by this method.

III. Experiments have been run on the equilibrium between postum ions, postum metal, arsenious acid, and arsenic metal in hydrochloric acid. From these preliminary results it is believed possible to accurately measure the potential of postum relative to arsenic. However, before the potential of postum can be determined, it will be necessary to obtain more accurate measurements on the potential of arsenic.

DETAILED REPORT

I. Bismuth-II Process - Stringham and Nelson

The equipment for the Bismuth-II process has been mounted and is ready for installation at Unit IV (Figure 1).

II. Investigation of Gamma Active Materials in the Aluminum Jackets - Rogers

The results previously obtained (Production Research, January 1-15, 1948, Section III) showed that, in addition to iron, a large portion of the gamma radiation from the aluminum jackets can be attributed to one of the following elements: zinc, cobalt, manganese, and nickel.

Classification changed to Unclassified
authority of Pub. David, etc. 6/10/90
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These elements can be separated from a solution containing aluminum by making the solution alkaline with sodium hydroxide and adding sodium peroxide. Zinc does not separate by this procedure when present alone, but is coprecipitated to a large extent when the other elements are present. Iron also separated with the foregoing elements.

Inactive zinc, cobalt, manganese, and nickel were added as carriers to an aluminum chloride solution which was obtained by dissolving the jacket of an active slug with hydrochloric acid. Separation of these elements by the preceding procedure separated 88 per cent of the gamma activity in a single precipitation.

The precipitate then was dissolved in hydrochloric acid and the active iron extracted (after adding inactive ferric chloride as a carrier) with isopropyl ether. It now remains to separate the remaining four elements to determine which will carry the gamma activity.

III. The Reduction of Postum from Bismuth Chloride Solutions - Kirby

In an effort to establish the potential of postum in hydrochloric acid solution experiments have been carried out on the equilibrium:

in which "m" is the positive valence of postum and "n" is the number of chlorine atoms in the postum complex.

Preliminary results indicate that equilibrium is reached at 25°C. after about 2 weeks of tumbling. The amount of postum remaining in solution after tumbling with arsenic in a saturated solution of arsenic (III) oxide in hydrochloric acid depends upon the acid concentration and the method of preparing the arsenic (Table I).

Table I
Reduction of Postum with Arsenic at 25°C.

Type of As	Conc. HCl, molal	Tumbling, days	Q in soln., d./min./ml.
vac. dist.	1.58	5	8.1×10^3
" "	"	7	4.6×10^3
" "	"	8	4.6×10^3
350°C.	1.707	1	0.5×10^3
"	"	11	0.3×10^3
"	"	22	0.5×10^3
100°C.	"	13	0.3×10^3
350°C.	4.547	2	10.14×10^4
"	"	11	1.92×10^4
"	"	22	0.95×10^4
100°C.	"	13	5.15×10^4
vac. dist.	4.75	8	2.56×10^6
" "	6.8	8	$(6.19 \times 10^7)^*$
" "	8.8	8	$(7.78 \times 10^7)^*$

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*This is essentially the concentration of the starting solution, hence no deposition occurred.

The "vac. dist." arsenic was prepared by subliming the metal at a few microns pressure. The "100°" and "350°" arsenic were prepared by subliming the arsenic in a current of dry nitrogen and then heating the sublimate in a stream of nitrogen for about fifteen minutes at 100°C. or 350°C., respectively.

Interpolation of the results to 4.5 molal hydrochloric acid shows that the different types of arsenic leave the following quantities of postum in solution:

vac. dist.	1.6×10^6 d./min./ml.
100°C.	4.6×10^4 d./min./ml.
350°C.	8.7×10^3 d./min./ml.

If the difference in potential between the various types of arsenic could be measured the valence of postum in the solution could be immediately calculated by the Nernst equation. However, it has not been found possible to measure the difference in potential because suitable electrodes could not be prepared. The difference in potential between the vacuum-distilled and 350°C. arsenic is calculated to be 33.5 or 67.0 millivolts if it is assumed that the valence of postum is 4 or 2, respectively.

The potential of postum in hydrochloric acid can be determined once the valence of postum and the potential of arsenic are known. Previous measurements of the potential of arsenic in hydrochloric acid (Production Research, December 16-31, 1947, Table II) differ by more than 100 millivolts from the results reported by Schuhmann, R., J. Am. Chem. Soc., 46, 1444-9 (1924).

In an effort to determine the potential of arsenic a study will be made of the equilibrium

with the various types of arsenic. The amount of arsenic remaining in solution when equilibrium is reached depends only on the hydrochloric acid concentration (and the type of arsenic) since all of the other components are present in fixed concentrations. The arsenic concentration, after equilibrium has been reached, calculated from the potential measurements of Schuhmann (I) and our measurements (II) are as follows:

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Conc. HCl, molal	Conc. HAsO ₂ molal	
	I	II
0.2	8.0×10^5	1.3
0.5	3.5×10^3	5.7×10^{-3}
1.0	3.7×10^{-1}	6.0×10^{-5}
2.0	1.6×10^{-3}	2.2×10^{-7}
4.0	8.5×10^{-3}	1.4×10^{-10}

From these calculations it can be seen that the difference in the measured potential has a tremendous influence on the calculated arsenic concentration.

FUTURE PLANS

Work is being started on the potential of tellurium in hydrochloric acid. Work will continue on the other problems now in progress.

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JHP/rcc

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