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SUMMARY

Fused Salt Processing

Dissolution rates of 3 to 4 mg/min·cm² have been obtained for specimens of STR elements in NaF-ZrF₄ using an air-lift type salt-metal contactor.

Fluorox

An activation energy of ~6 kcal/mole has been calculated for the reaction 2UF₄ + O₂ → UF₆ + UO₂F₂ over the temperature range 200-800°C.

Neptex

Three kilograms of K-25 fluorination ash containing ~150 mg of Np are being processed in the 3/4" pulse columns to demonstrate the modified Neptex flowsheet.

Excer

The presence of a trace of sodium ion in the Excer cell feed caused the production of high density UF₄.

Feed Materials

Metallex

Packed column washing of thorium amalgam is five times faster than batch stirring methods, but thorium losses were 6.66%.

Vacuum drying thorium amalgam in a packed column is also faster and produces a metal with a ThO₂ content comparable to small batch vacuum drying.

Uranium chloride from Zircex-processed U-Zr alloy was reduced to
UHg₄ by Metallex with a yield of $\sim 80.5\%$.

**Hermex**

The feasibility of continuous mercury distillation from cold-pressed uranium amalgam compacts and melting of the product uranium was demonstrated.

**Heterogeneous Reactor Fuel Reprocessing**

The chemical removal of zirconium cladding from a PWR fuel element containing a U-Mo alloy core was demonstrated in the laboratory. Over 99% of the uranium was obtained in a water solution separated from the insoluble molybdic acid.

**Volutility Studies**

The true solubility of NiF₂ in NaZrF₅ appears to be less than 1 weight percent at 600°C. At 1 weight percent and higher concentrations the NiF₂ apparently is in the form of a fairly stable dispersion or colloid.

**Mass-233 Studies**

Over 90% of the protactinium from spiked simulated Thorex aqueous waste adsorbs on preformed zirconium phosphate. The protactinium also carries completely on thorium precipitated from this solution with NaOH.

The following equation holds for the solvent extraction of Pu⁴⁺ with 5% TBP: $\log DC O/A = 2[Ai(NO₃)₃] - 1.3$.

**Homogeneous Reactor Blanket Processing**

Adsorption of plutonium on titanium, Zircaloy-2, and type 347 stainless steel at 250°C from 1.4 m UO₂SO₄ containing 0.5 to 7.0 mg Pu/kg H₂O for exposure times of 15 to 160 hr may be expressed by the
following equations:

\[ A(Tt) = 0.022 \, C^{2/3} \, t^{1/3} \]
\[ A(Zr-2) = 0.063 \, t^{1/3} \]
\[ A(347 \, SS) = 0.0082 \, t \]

Where \( A \) is adsorption in \( \mu g/cm^2 \), \( C \) is plutonium concentration in \( mg/kg \, H_2O \), and \( t \) is exposure time in hours. Standard deviation for duplicate experiments is about 50%.

Homogeneous Reactor Fuel and Blanket Studies

Viscosity measurements and radiographs at 100-300°C on a slurry from loop run 200A-5 at Y-12 indicate that the pumping troubles observed at 200°C are associated with marked changes in the slurry characteristics occurring at this temperature. The effect of MoO_3 catalyst concentration on the gas recombination rate is being investigated. Slurries of thorium-uranium mixed oxides give lower specific conductances than do pure thorium oxide slurries. The differences in pH observed between thorium slurries and their supernatants appear associated with the glass electrode and disappear with increasing oxide calcination temperature.
Further experiments were performed to elucidate the effects of temperature and HF flow rates on the dissolution rate of STR fuel elements in molten NaF-ZrF₄ eutectic. The melt was agitated, and intimate contact of the agitated melt with the STR element pieces was achieved by placing the pieces in the center pipe of an air-lift type pump so that the rising gas-liquid mixture passed over them.

A summary of results obtained is given in the table below. Dissolution rates are based on specimen weight losses.

<table>
<thead>
<tr>
<th>No.</th>
<th>Temperature (°C)</th>
<th>HF Flow Rate (mg/min)</th>
<th>Dissolution Rate (mg/min·cm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>600</td>
<td>25</td>
<td>2.4</td>
</tr>
<tr>
<td>2</td>
<td>600</td>
<td>47</td>
<td>4.5</td>
</tr>
<tr>
<td>3</td>
<td>600</td>
<td>90</td>
<td>3.4</td>
</tr>
<tr>
<td>4</td>
<td>600</td>
<td>90</td>
<td>2.6</td>
</tr>
<tr>
<td>5</td>
<td>800</td>
<td>25</td>
<td>3.4</td>
</tr>
<tr>
<td>6</td>
<td>800</td>
<td>47</td>
<td>4.5</td>
</tr>
<tr>
<td>7</td>
<td>800</td>
<td>90</td>
<td>5.7</td>
</tr>
</tbody>
</table>

It will be observed that, in general, the rates at 800°C are greater than those at 600°C at a given HF flow rate. Experimental errors are quite large, however (compare experiments 3 and 4), so a critical comparison is not possible. Interestingly, the dissolution rates at 600°C appear to pass through a maximum at a HF flow rate of 47 mg/min. The rates at 800°C exhibit no such effect.

It should be noted that these dissolution rates are all 5 to 10 times those obtained previously when the HF gas was simply introduced.
(at comparable flow rates and melt volumes) into the melts through a piece of nickel tubing. This observation emphasizes the profound influence of a change in method of HF introduction, and suggests that large scale dissolutions will need to be performed before a practical dissolution rate can be estimated any closer than a factor of two.

2.0 FLUOROX
(R. G. Wymer)

2.1 Kinetics of UF₄ Oxidation (L. M. Ferris)

In the February 10 weekly report the rate equation

$$\frac{-d(UF_4)}{dt} = kA_pO_2$$

(1)

was presented for the reaction $2UF_4 + O_2 \rightarrow UF_6 + UO_2F_2$. In evaluating a specific reaction rate constant from equation (1) it must be assumed that $A$, the total surface area of the $UF_4$, remains constant which, of course, is not the case since $UF_4$ is continually being used up in the reaction. A more correct rate equation,

$$\frac{-d(UF_4)}{dt} = kS(UF_4)pO_2$$

(2)

is derived from equation (1) by assuming that the specific surface area ($S$, $\text{meters}^2/\text{g}$) is independent of temperature and time. In equation (2), $(UF_4)$ is the number of moles and $M$ is the molecular weight of $UF_4$ and $pO_2$ is the partial pressure of oxygen. Using equation (2), $k$ is found to be $6.3 \times 10^{-5}$ moles/min-atm-meter² at 800°C. An energy of activation of $\sim 6$ kcal/mole is obtained for the temperature range 200-800°C.
3.0 NEPTEX
(J. R. Flanary and J. H. Goode)

3.1 Recovery of Np\textsuperscript{237} from K-25 Fluorination Ash

The following revised flowsheet has been developed for the dissolution of K-25 fluorination ash (mixed uranium fluorides) and the recovery of Np\textsuperscript{237} by solvent extraction from the resulting feed solution. Basis: 1 kg ash.

A. Dissolution

1. Slurry 1 kg ash in 2.67 liters Diban.

2. Add 4.5 kg ANN, 1.47 liters 16 Mö HNO\textsubscript{3}, dilute to 13 liters with water. Heat to 60-70°C to increase dissolution rate. Sample.

3. Make final feed adjustment and make 0.01 M in ferrous sulfamate to reduce neptunium.

B. Solvent Extraction

\begin{align*}
\text{AF}/100 & \text{ volumes: } 50 \text{ g U/l, } 1.8 \text{ M Al, } 1 \text{ M HNO}_3, 0.01 \text{ M } \text{Fe(NH}_2\text{SO}_3)_2, \\
& 3-5 \text{ mg Np/l} \\
\text{AS}/20 & \text{ volumes: } 0.75 \text{ M Al, } 0.5 \text{ M HNO}_3, 0.01 \text{ M } \text{Fe(NH}_2\text{SO}_3)_2 \\
\text{AX}/150 & \text{ volumes: } 15\% \text{ TBP in Amsco 125-82} \\
\text{BS}/60 & \text{ volumes: } 15\% \text{ TBP in Amsco 125-82} \\
\text{BX}/40 & \text{ volumes: } 0.5 \text{ M HNO}_3 \\
\text{CX}/210 & \text{ volumes: } 0.01 \text{ M HNO}_3
\end{align*}

Results of batch countercurrent experiments indicate that approximately 2 and 5 extraction stages are required to obtain losses of less than 0.01% for uranium and neptunium, respectively. About 4 back-extraction stages are needed to hold the uranium loss to the neptunium product from the partitioning column to less than 0.01%; 5 stages are required for the neptunium-uranium partitioning.
The neptunium product (EP) will contain about an equal weight of uranium ($\sim 10$ mg/l), and is to be concentrated and isolated by ion exchange techniques.

Three kilograms of ash have been dissolved to make 40 liters of feed, which is being processed in the 0.75 inch diameter pulsed column facility in an extended demonstration run.

$^4.0$ EXCER
(I. R. Higgins)

Mallinckrodt has offered to evaluate the bomb reduction characteristics of the Excer UF$_4$ product. Five pound samples were required, so ion exchange runs were interrupted to prepare more high density products. We were faced again with the problem of what were the density controlling factors. A feed was made up similar to one which gave a density of 3.4 previously by digesting with a few grams of UF$_4$ per liter. Tap density of product was 2.2. It was then thought to be a function of current density and a reduction was made at 1 amp/in$^2$ instead of 2. Tap density of product was 1.4. There was a possibility that the available fluoride influenced crystal formation. A 10% deficiency instead of 10% excess of HF was added. Tap density of product was 2.4. The first high density product obtained had 0.01 g/l of Na in the feed. A pinch of salt was added to the feed which had just delivered a tap of 2.4. The final tap density was 2.7.

5.0 FEED MATERIALS
(O. D. Dean)

5.1 Metalllex (O. C. Dean)

5.1.1 Countercurrent, Packed Column Amalgam Washing

One and four tenths percent thorium amalgam was washed with
3 HCl passes both countercurrently and in a 1-1/2 in. i.d. x 6 in. glass bead-packed spray column. The washed amalgam was vacuum dried by one pass in another similarly packed column, filtered without air exposure, pressed and vacuum-sintered.

Countercurrent column washing and drying performs the operation ∼ 5 times as fast as batch-handling. Flows through the wash column were erratic, due to the insufficient gravity head used. There was excessive amalgam holdup due to the use of glass wool as a packing support.

Thorium losses in washing were 6.66%. Improvement is expected from correction of flow rates and amalgam holdup. The final sintered metal had a good appearance, a density of 10.5 g/cc and 0.66% ThO₂ content. The ThO₂ content expected from non-vacuum dried amalgam is ∼ 1.36% and for batch-vacuum dried amalgam ∼ 0.7%.

5.1.2 Reduction of Zircex Uranium Chloride

Nine and five tenths grams of uranium chloride resulting from hydrochlorination of 2% U-Zr alloy which was Zircaloy-clad was reduced in the Metallex Mark II reactor with 170 ml of 3.3 M Na amalgam. The reaction was carried out at 200°C at 2000 rpm for 30 min. The amalgam was washed with 1 N sulfuric acid and deaerated water and filtered. Thirty-eight grams of uranium quasi were recovered, containing 1.97 grams, or 35% of the U metal. The U losses from the washing procedure were only 19.5%. If this number can be accepted as a base for calculation, the reduction yield was 80.5%. The material balance was only 60%, and the U analysis of the quasi-amalgam is in question.

5.2 Hermex (E. Sturch)

5.2.1 Furnacing Pressed Cylinders of Uranium Amalgam

To test the possibility of continuous distillation of mercury
from U quasi-amalgam and melting of the U product, quasi prepared by dissolution of uranium in boiling mercury was pressed into 12 cylinders approximately 1 in. in length and 1/2 in. in diameter and containing ~15% U. These cylinders were stacked in a 5/8 in. quartz tube perforated to within 6 in. of the bottom. The mercury was boiled off and the uranium melted and recovered as a compact slug of metal.

Boiling mercury dissolution of uranium gave a thick amalgam which was concentrated by pressing in a 1/2 in. die. These quasi cylinders retained their shape when stacked in a perforated quartz tube and heated slowly to 800°C, at which point they appeared to have lost most of their residual mercury. The temperature was increased to 1400°C, and after 4 hours at this temperature, followed by slow cooling, a compact slug of metallic uranium was obtained.

This furnacing technique depends upon the progressive fusion of the quasi cylinder into a pool of molten uranium at the bottom of the tube in the hot zone of the furnace. The column of cylinders moves down the tube as the bottom cylinder melts into the molten uranium. The hold-up in the upper part of the tube is long enough for the mercury to vaporize out of the cylinders in the cooler zone of the furnace before they reach the hot zone and the pool of molten uranium.

6.0 HETEROGENEOUS REACTOR FUEL REPROCESSING
(J. E. Savolainen)

6.1 Zirconium Cladding Removal by Hydrochlorination (J. E. Savolainen)

The removal of zirconium cladding by hydrochlorination from a uranium-molybdenum alloy core was demonstrated on laboratory scale. A 23.79 g section of PWR fuel element was hydrochlorinated for 3-1/4 hr at 330°C. All of the zirconium cladding, 4.56 g, was removed. A small amount, 5.5% of the core alloy, also was hydrochlorinated. The remainder of the core metal was then dissolved in 10 M HNO₃ in 2 hr and 10 min.
The dissolution of the metal was complete even though insoluble molybdic acid formed during the dissolution.

The dissolver solution was filtered to separate the molybdic acid. The filtrate and washes combined contained 94.5% of the total uranium at a concentration of 71.4 g/l. The uranium chlorides, which can be converted to nitrates, and the filtered dissolver solution contained 99.9% of the total uranium.

The molybdic acid filter cake was dissolved in 4 M NaOH and contained 0.04% of the uranium after filtering and centrifuging. A small amount of uranium, 0.036% of the total, was retained by the fritted glass filter which was also used to remove the molybdic acid from the dissolver solution, while 0.004% was recovered as a fine precipitate by centrifugation of the sodium molybdate solution.

The amount of uranium converted to the chloride during jacket removal could be reduced by using a more inert core alloy. The alloy, 10-12% Mo-U, used in this demonstration has a hydrochlorination rate of 0.49 mg/cm²/min. A 0.2% Mo-U alloy has a hydrochlorination rate of 0.095 mg/cm²/min while that of a 0.5% Si-U alloy is 0.057 mg/cm²/min.

7.0 VOLATILITY STUDIES
(G. I. Cathers)

7.1 Fused Salt Tests (M. R. Bennett)

Based on qualitative and visual observation of the behavior of NiF₂ in molten NaF-ZrF₄ (50-50 mole percent), the solubility of NiF₂ at 600°C is less than 1 weight percent. NiF₂ in excess of the solubility acts as a colloid. Lowering or raising the temperature at a given nickel concentration leads to the determination of a "cloudpoint" or "clearpoint" respectively, since the solubility temperature coefficient is apparently positive. The NiF₂ colloid formed in NaF-ZrF₄ appears fairly stable up to concentrations of several percent. These
observations are necessarily tentative since they were made with crude apparatus where hydrolysis from moisture in the air was a complicating factor.

The formation of a major amount of NiF₂ sludge has been encountered in only two laboratory tests to date. In both cases the NaF-ZrF₄ salt had been subjected to fluorination treatment prior to the hydrofluorination period in which the sludge first became noticeable. This has suggested that perhaps a large amount of NiF₂ had accumulated as a fairly stable suspension during fluorination, and that hydrofluorination then resulted in the coagulation of this material and sludge formation.

Attempts to study the solubility behavior of NiF₂ in molten NaF-ZrF₄ by (1) periodic additions of NiF₂ or (2) forming NiF₂ in the salt by prolonged fluorination have proved unsuccessful mainly as a result of inconsistent analytical data. This has possibly been due to the non-homogeneous character of the melts, and to the fact that samples were taken after allowing the material to stand for a few minutes.

8.0 MASS-233 STUDIES
(R. H. Rainey)

8.1 Protactinium Recovery (A. B. Meservey)

Studies are continuing on the removal of tracer quantities of Pa from simulated Thorex A-column waste. These experiments will be verified using pilot plant solutions as soon as active solutions are available. Adsorption curves for Pa on preformed zirconium phosphate having 1.5% the volume of the solution reveal an adsorption of better than 99.5% in a pH range of 1.0 to 2.8. Only 92% of the Pa adsorbs from simulated APC in the same pH range.

When the aqueous waste containing 1.8 M aluminum and 7 g Th/l was made alkaline with NaOH, about 99.99% of the Pa adsorbed on the
thorium precipitate. Adsorption of Pa on zirconium phosphate was more efficient from the sodium aluminate solution than from the acid solution.

8.2 Solvent Extraction of Plutonium (R. G. Mansfield)

The tetravalent plutonium distribution coefficient of 5% TBP in Amsco as a function of aluminum nitrate salting strength gives a curve closely approximated by the equation:

$$\log DC/DA = 2[Al(NO_3)_3] = 1.3$$

The DC increases from about 0.1 at 0.15 M $\text{Al(NO}_3\text{)}_3$ to 0.5 M HNO$_3$ to 200 at 1.8 M $\text{Al(NO}_3\text{)}_3$--0.5 M HNO$_3$. Using this range of distribution, the extraction conditions of plutonium are being adjusted to give maximum decontamination in the Thorex solvent extraction columns consistent with a 1% plutonium loss.

9.0 HOMOGENEOUS REACTOR BLANKET PROCESSING (R. E. Leuze)

9.1 Plutonium Adsorption on Metals (J. M. Chilton)

After tabulating all recent data for plutonium adsorption on metals at 250°C from 1.4 m $\text{UO}_2\text{SO}_4$ containing 0.5 to 7.0 mg Pu/kg H$_2$O, the following was observed:

1. Plutonium adsorbed on all metals tested. The amount adsorbed was different on each metal and increased in the following order: gold, titanium, Zircaloy-2, and type 347 stainless steel.

2. Less plutonium adsorbed on bright, clean metal surfaces than on preconditioned surfaces or metals covered with an oxide film.

3. There was no evidence of equilibrium for exposure times up to 160 hr.

The most consistent adsorption results were obtained on bright, clean titanium metal. Adsorption at 250°C from 1.4 m $\text{UO}_2\text{SO}_4$ containing
0.5 to 7.0 mg Pu/kg H₂O for exposure times of 15 to 160 hr can be represented by the following equation:

\[ A(T) = 0.022 \cdot \frac{C^2}{3} \cdot t^{1/3} \quad (1) \]

where \( A \) is the plutonium adsorption in µg/cm², \( C \) is the plutonium concentration in mg/kg H₂O, and \( t \) is time in hours. This equation must be used with caution since the standard deviation of duplicate determinations was about 50%.

Plutonium adsorption on Zircaloy-2 was less consistent than adsorption on titanium. Unfortunately, the variation in results from duplicate runs was so great that the effect of plutonium concentration between 0.5 and 7.0 mg/kg H₂O could not be determined. For the same conditions as equation (1), the plutonium adsorption on Zircaloy-2 may be expressed as:

\[ A(Zr-2) = 0.063 \cdot t^{1/3} \quad (2) \]

with a standard deviation of about 50%.

Less data are available for plutonium adsorption on stainless steel. As with Zircaloy-2 the variation in duplicate experiments was too great to determine the effect of plutonium concentration between 0.5 and 7.0 mg/kg H₂O. The data indicate that adsorption increases linearly with time. For the same conditions as equation (1), plutonium adsorption on type 347 stainless steel may be expressed as:

\[ A(347 SS) = 0.0082 \cdot t \quad (3) \]

with a standard deviation of about 50%.

10.0 HOMOGENEOUS REACTOR FUEL AND BLANKET STUDIES
(J. P. McBride)

10.1 Studies on 200A-5B-10L #2 (N. A. Krohn)

Viscosity measurements were made in the dash pot irradiation bomb on slurry sample 200A-5B-10L #2 using the Thyratron timer. The sample
was that of a slurry undergoing engineering test at Y-12 and contained 1200-1500 ppm of sulfate. The sample as received had a concentration of about 600 g Th/kg H₂O. The viscosity at this concentration did not change appreciably up to temperatures of 200°C, having an average value of 10.5 centistokes. At similar concentrations 900°C calcined D-16 has a viscosity of 4.3 centistokes and pumped 900°C D-16 (X-28), 7.0 centistokes. At 250 and 300°C it was impossible to maintain the 200A-5B-10L #2 material in suspension except at frequencies around 4 cycles per second. As the frequency was lowered the apparent viscosity increased considerably.

Radiographs of the bomb at 300°C showed a settled bed in the bottom of the bomb which could not be suspended by the action of the stirrer. After stirring at 100 and 200°C and settling 60 sec at each temperature, radiographs showed a slurry plug on top of the stirrer similar to those obtained consistently with pure ThO₂ slurries at 200°C and once at 100°C.

In the engineering test at Y-12 pump power requirements were found to become erratic and fluctuate around 200°C. Considering the above results, this trouble seems to be associated with marked changes occurring in the physical characteristics of the slurry at this temperature.

10.2 Gas Recombination Studies (L. E. Morse)

Studies on the effect of MoO₃ catalyst concentration on the combination rate of stoichiometric H₂-O₂ mixtures in 900°C recalcined D-16 ThO₂-water slurries containing 1000 g Th/kg H₂O and 0.5% U were extended to 0.15 M MoO₃. Increasing the MoO₃ concentration from 0.10 to 0.15 M increased the hydrogen consumption from 0.8 moles H₂ per hour per liter of slurry at 276°C to 6.8 moles H₂ per hour per liter of slurry at 279°C, both rates calculated for a partial pressure of 500 psi H₂.
Treatment of the slurry with hydrogen (1000 psi at 25°C) for 1.2 hr at 280°C did not increase the activity of the catalyst in the slurry containing 0.15 m MoO₃ to the degree reported previously for a slurry containing 0.10 m MoO₃ treated in a similar manner. The combination rate of the stoichiometric gas mixture after "hydrogenation" of the slurry containing 0.15 m MoO₃ catalyst was equivalent to 8.8 moles H₂ per hour per liter of slurry at 243°C and a partial pressure of 500 psi H₂. The rate was not increased by further treatment with H₂ for 3 hr at 280°C. Subsequent treatment of the slurry with oxygen (500 psi at 25°C) for 2.5 hr lowered the hydrogen consumption to 6.2 moles H₂ per hour per liter of slurry at 253°C and a partial pressure of 500 psi H₂.

Experiments at high MoO₃ are now in progress to determine if the behavior of the slurry containing 0.15 m MoO₃ is typical for slurries with concentrations of MoO₃ higher than 0.1 molal. Experiments at 0.05 m MoO₃ are also being carried out.

The slurries were prepared by tumbling the dry ThO₂, UO₃·H₂O, and MoO₃ powders for 1 hr and then heating the mixture in H₂O at 280°C for 3 hr in the presence of oxygen (300 psi at 25°C). The MoO₃ was prepared by heating ammonium paramolybdate at 480°C for 18 hr.

10.3 Conductivity Measurements with Thorium-Uranium Oxide Slurries (C. E. Schilling)

The specific conductance of slurries of several thorium-uranium oxides containing 0.37-0.57 mole % U (based on Th) has been measured at 29°C and a concentration of 175 g Th/kg H₂O. Slurries of mixed oxides prepared by wet-autoclaving (300°C) a simple mixture of the oxides and reirling the product at 900°C gave specific conductances only slightly lower than slurries of the pure oxide. Slurries of mixed oxide prepared by the hydrothermal or thermal decomposition of the coprecipitated tetravalent uranium-thorium oxalates gave much lower conductances.
Under the conditions of the measurement a slurry of D-16 oxide calcined at 800°C gave a specific conductance of 13.0 reciprocal micro-ohms per cm. The slurries of the mixed oxides prepared from the simple mixture and containing 0.5% U had conductances of 11 reciprocal ohms per cm. Slurries of mixed oxides prepared from the coprecipitated oxalates and subsequent thermal or hydrothermal decompositions containing 0.37 to 0.57% U showed specific conductances varying between 2 and 6 reciprocal ohms per cm. No trend of conductance values with U concentration was noted.

10.4 Slurry pH Measurements (E. V. Jones)

Systematic pH measurements have been carried out on slurries of pure thorium oxide, and oxide slurries containing sulfuric acid, sulfuric acid plus 0.005 M Na_2P_2O_7, and sulfuric acid plus 0.005 M Na_2SiO_3 over the sulfate concentration range of 0 to 20,000 ppm. A glass electrode and a saturated calomel reference electrode were used in making the measurements. Four measurements were made on each slurry: (1) with both electrodes in the slurry; (2) with both electrodes in the supernatant with the solids settled out; (3) with the glass electrode in the settled slurry and the reference electrode in the supernatant; and (4) with the glass electrode in the supernatant and the calomel electrode in the settled slurry.

At slurry pH's above 5 the pH measurement with the glass electrode in the slurry (positions 1 and 3) were 0.2 to 2 pH units higher than those obtained with the glass electrode in the supernatant (positions 2 and 4) regardless of the position of the calomel reference electrode. Hence the discrepancies between the pH of the slurry and the supernatant are associated with the glass electrode. In general, the difference in pH between the slurry and supernatant decreased with oxide calcination temperature disappearing altogether for the 1000°C material.
In the presence of $H_2SO_4$ and using the 900°C material, the pH difference remained relatively constant at 0.3 for sulfate concentrations of 0 to 1000 ppm, increased to 0.75 at 2500 ppm sulfate (slurry pH = 8.0), and decreased to 0 at 5000 ppm sulfate. With 0.005 M $Na_2P_2O_7$ present along with the $H_2SO_4$, the pH difference decreased from 1 to 0.35 as the sulfate increased from 0 to 1000 ppm, increased to 1.2 at 2500 ppm sulfate (slurry pH = 9.2), and decreased to 0 at 5000 ppm sulfate. With 0.005 M $Na_2SiO_3$ and the 800°C material the pH differences were more than twice as great for a given sulfate concentration and showed the same general trends but the data is not complete enough to indicate whether or not a maximum in the pH difference occurs around pH 8-9 as in the previous two studies.

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