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PURIFICATION OF STRONTIUM-90 FROM MAGNESIUM

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PURIFICATION OF STRONTIUM-90 FROM MAGNESIUM

by

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March 15, 1965

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INTRODUCTION

Although purification from magnesium is not obtained in the D2EHPA (Strontium Semiworks) strontium purification process, this has not been a matter of concern in the past for two reasons: 1) magnesium is removed in the lead sulfate precipitation^(1,2) process (magnesium sulfate is highly soluble) and 2) there is very little magnesium in the uranium metal or other essential materials currently used. Both situations are expected to change. The lead carrier process will not be used in the Waste Management Program⁽³⁾ to process current wastes and proposed alterations in the metallurgical processes used by the uranium suppliers may result in the introduction of significant quantities of magnesium. Its presence in the purified strontium used for a heat source would dilute the fuel, lower the specific power and may cause undesirable changes in the physical and chemical properties of the fuel. Separation techniques were accordingly sought and a process was developed to eliminate magnesium.

SUMMARY

The D2EHPA solvent extraction behavior of magnesium was measured and found to be identical to that of strontium⁽⁴⁾. Addition of complexing agents (such as DTPA) also failed to result in a change in the relative extractabilities of the two, precluding magnesium separation by this scheme.

A precipitation process, utilizing low solubility of magnesium hydroxide was successfully developed to eliminate magnesium. This process

may also be applicable to the final purification step⁽³⁾ (eliminating the proposed strontium purification using D2FHPA solvent extraction), since calcium, cerium, iron and rare earths are also precipitated away from the strontium fraction. Less than 5 per cent of the magnesium and essentially none of the iron and cerium initially present in the synthetic feed remained in solution at pH 10. Less than 2 per cent of the initial strontium was lost below a pH of 11, but the loss increased to 15 per cent at a pH of 13. Approximately 98, 51 and 8 per cent of the calcium remained in solution as a function of the respective pH values of 11, 12.4 and 13. The final precipitate occupies a volume less than 8 per cent that of the original solution and is somewhat gelatinous, but centrifuges well.

EXPERIMENTAL

A crude strontium feed composition⁽³⁾, for the D2EHPA purification of strontium from current acid wastes, is shown in Table I.

Table I

<u>Crude Strontium Feed⁽³⁾</u>	
Sr ⁺⁺	0.058 <u>M</u>
Mg ⁺⁺	0.017
Ca ⁺⁺	0.012
Fe ⁺⁺⁺	0.01
Na ⁺	0.2
Cr ⁺⁺⁺	0.001
R. E.	0.002
H ⁺	0.12

To determine the conditions for hydroxide precipitation, samples

of the feed were heated to 80 - 100° C and the pH adjusted with 18.5 M NaOH. The resulting slurry was then digested for 5 minutes and centrifuged. The supernate was sampled for pH and component determinations. The major components of the feed were traced as a function of pH by use of radio strontium-85 (purchased from Abbott Laboratories), magnesium-28 (purchased from Brookhaven National Laboratory), and cerium-144 and iron-59 (purchased from Oak Ridge National Laboratory). Calcium measurements were determined by flame photometry.

To further understand the hydroxide precipitation of the crude strontium feed, a one liter portion was titrated with 18.5 M NaOH using a Metrohm, Model E-336, recording potentiograph.

DISCUSSION

Magnesium distribution ratios were determined for 0.4 M di (2-ethylhexyl) phosphoric acid (D2EHPA) -- 0.2 M tributyl phosphate (TBP) and were found to be identical to the previously reported strontium results⁽⁴⁾. Therefore, magnesium will follow strontium in the solvent extraction process proposed for the Waste Management - Fission Product Purification Program⁽³⁾. At the present time, any magnesium initially present is probably separated from the strontium during the lead sulfate precipitation process (MgSO_4 solubility: 73.8^{100} grams/100 ml H_2O and SrSO_4 , 0.0114^{30} grams/100 ml $\text{H}_2\text{O}^{(5)}$)(1,2). But in Phase III of the Waste Management program, the acidic waste will go directly to the solvent extraction process and a magnesium-strontium separation may be required during strontium product purification.

It was proposed that a hydroxide precipitation would separate magnesium from strontium and it might also be possible to remove calcium, iron, cerium and rare earths at the same time. If so, the present plans for a strontium solvent extraction final purification step⁽³⁾ could be eliminated.

Conditions for the hydroxide precipitation are shown in Figure 1. All of the cerium and iron precipitated above a pH of 9. Less than 5 per cent of the magnesium remained in solution above a pH of 10. Less than 2 per cent of the strontium was lost below a pH of 11. Above a pH of 11, strontium losses increase to 15 per cent at a pH of 13. Conditions for a calcium - strontium separation will necessitate going to a pH of 13 to remove > 90 per cent of the calcium with an associated 15% loss of strontium. The precipitate waste, normally thrown away, could then be washed to solubilize the strontium remaining in the precipitate or several batches of precipitate could be collected prior to dissolution and reprecipitation.

To further define the relationship between the hydroxide requirements for precipitation and the pH at which various components of the crude strontium feed would precipitate, a synthetic sample was titrated as shown in Figure 2. The results show clearly that magnesium precipitates at ~ pH 10, prior to strontium and calcium.

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FIGURE 1

Magnesium-Strontium Separation-Hydroxide Precipitation

FIGURE 2

Titration of a Strontium-Magnesium Feed

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