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# Scrubbing of Iodine from Gas Streams with Mercuric Nitrate—Conversion of Mercuric Iodate Product to Barium Iodate for Fixation in Concrete

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## ABSTRACT

A bench-scale model of a mercuric nitrate scrubber for removal of iodine from off-gas streams was constructed and operated in conjunction with a mercuric iodate—to—barium iodate conversion system to determine the feasibility of total recycle of all processing solutions. The two main aspects of the system examined were (1) the extent of contamination of the barium iodate product, and (2) the effect of cross-contamination of various process solutions on the efficiency of the process.

The experimental evidence obtained indicates that, with appropriate control, all solutions can be recycled without significant contamination of the product that would be harmful to the host concrete or to the environment. Mercury contamination was found to be $\leq 0.5$ wt % of the barium iodate product. The most significant effect on system efficiency was determined to be barium hydroxide contamination of the sodium hydroxide solution used to convert mercuric iodate to sodium iodate. A mole ratio of barium hydroxide to sodium hydroxide of about 1:225 caused a decrease in conversion efficiency of about 45%.

## 1. INTRODUCTION

The waste management of fission product iodine requires special attention because of its high biological significance. Fortunately, most of the iostopes have short half-lives. However, $^{129}I$ has a half-life of $1.59 \times 10^7$ years and must be permanently isolated from the biosphere. Laboratory studies demonstrated that this isotope can be fixed as $Ba(IO_3)_2$ and incorporated in concrete for permanent disposal.[1-4]

One of the proposed methods for removing iodine from off-gas streams is a mercuric nitrate—nitric acid scrubbing system. This scrub solution reacts with both elemental and organically combined forms of iodine to yield mercuric iodate.[2-6] Although mercuric iodate is a very insoluble compound, mercury compounds are sources of accumulative heavy-metal poisons and are considered environmental hazards if released to the biosphere. Barium iodate is less toxic to the environment and can be readily incorporated into concrete mixes, and the result is a decreased environmental hazard. Thus, studies were initiated to find an acceptable method for converting mercuric iodate to barium iodate. To be acceptable, the conversion process must meet the three following criteria:

1. All solutions used in the system must be recycled to avoid creating additional waste streams.
2. The mercury content in the barium iodate product must be minimal to avoid additional environmental hazards.
3. Cross-contamination must be minimized to ensure high conversion efficiency.

This report describes the results of an investigation made using a bench-scale model of a mercuric nitrate—nitric acid iodine scrubbing system in combination with a mercuric iodate—barium iodate conversion system. Operating parameters were adjusted to determine the conditions that would result in meeting the above criteria.

## 2. EQUIPMENT AND PROCEDURES

A bench-scale model of a mercuric nitrate—nitric acid iodine scrub system and mercuric iodate—to—barium iodate conversion system was assembled as shown schematically in Figure 1. The equipment was primarily borosilicate glass with glass/Teflon valves and some interconnections of heavy-wall Tygon tubing. Teflon tape was used on vessel stoppers to avoid use of stopcock grease. The scrub column was a 1-in.-ID glass column filled to a depth of 36 in. with perforated, extruded stainless steel packing. The storage capacity for mercuric nitrate—nitric acid solution was about 1.3 ℓ at the bottom of the scrub column and in the adjacent reservoir. The components were arranged for gravity flow wherever possible, and Teflon bellows pumps were used to transport the solutions. Solution reservoirs and reaction vessels were equipped with heating mantles or tape heating elements. Solids were separated from the liquids by vacuum filtration through fine-porosity fritted glass filters.

In this system, air containing gaseous iodine was passed countercurrently through a mercuric nitrate—nitric acid solution. This converted the iodine to mercuric iodate, which remained in the solution. The solution was oxidized by partial evaporation to obtain the iodate.[3] The mercuric iodate product was converted to barium iodate in a two-step process:

$$Hg(IO_3)_2 + 2\ NaOH \longrightarrow HgO + H_2O + 2\ NaIO_3\ ,$$

$$2\ NaIO_3 + Ba(OH_3) \longrightarrow Ba(IO_3)_2 + 2\ NaOH.$$

The mercuric oxide was filtered from the sodium iodate solution, reacted with nitric acid to form mercuric nitrate—nitric acid solution, and was returned to the iodine scrub system. In the final step, sodium iodate solution was reacted with barium hydroxide solution to form barium iodate and sodium hydroxide. The barium iodate was filtered out for final storage in the waste form, and the sodium hydroxide was recycled. Thus, all solutions were recycled and no additional radioactive waste that would require further treatment or storage was generated.

The scrub system operating conditions were selected, based on previous experimentation, to produce at a reasonable efficiency a maximum amount of product per time period.[3,4,6-9] From previous studies, the following operational conditions were selected:

Column scrub solution - 0.4 $\underline{M}$ $Hg(NO_3)_2$; 14 $\underline{M}$ $HNO_3$

Column solution flow rate - 40 ml/min

Gas (dry air—$I_2$) - 5 ℓ/min

Iodine feed rate - 0.2 g/h

Conversion system solutions - 1 $\underline{M}$ NaOH and $\sim$2 $\underline{M}$ $Ba(OH)_2$

After the scrub column had accumulated about 25% of its iodine capacity ($\geq$12 g/ℓ), about 50% of the scrub solution was routed to the evaporator for iodate conversion. The solution volume was reduced about 60% by evaporation. The distillate was returned to the scrub solution reservoir, and the mercuric iodate solution was dropped to a reaction vessel where the mercuric iodate was allowed to settle out during completion of the

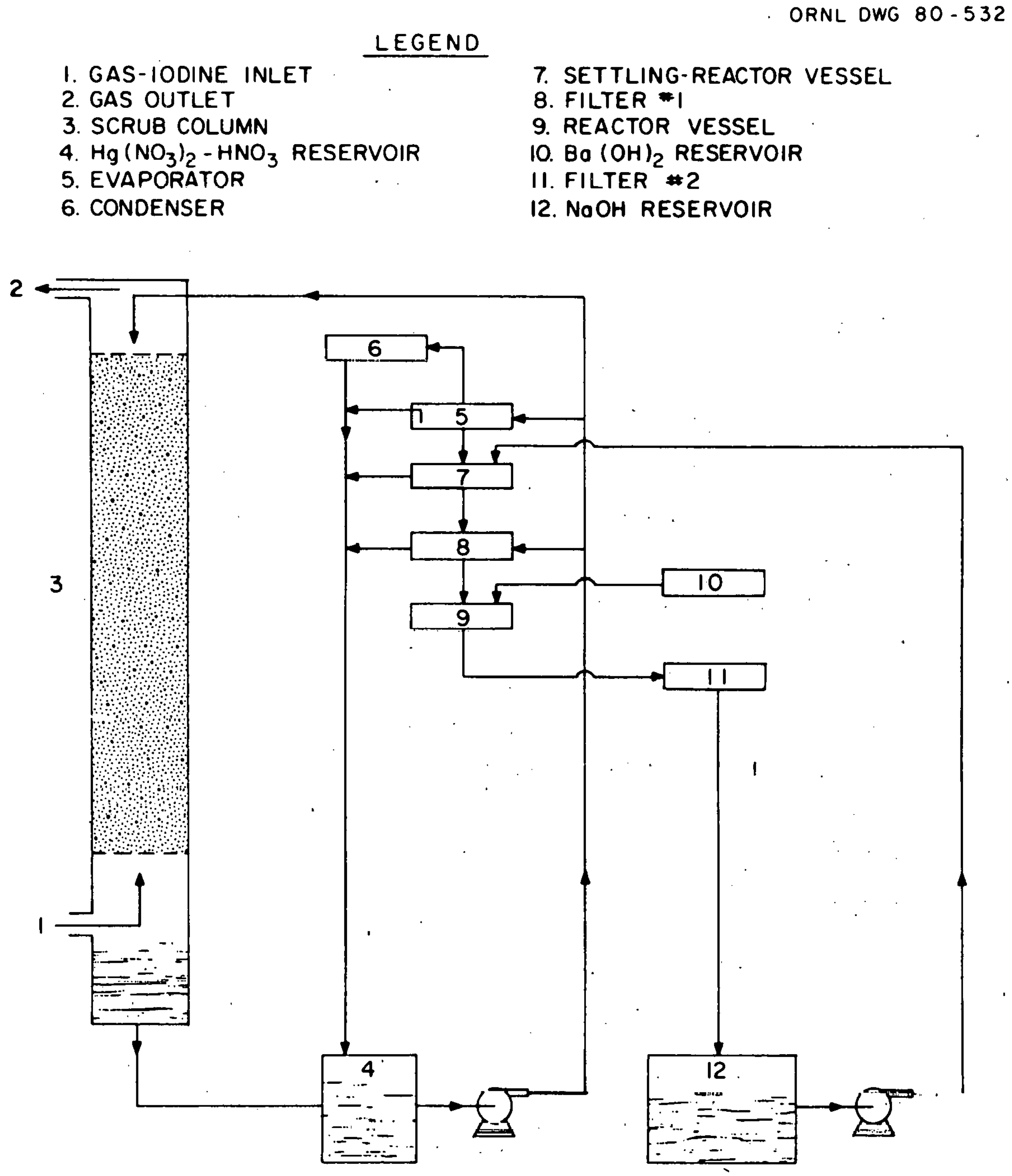

Fig. 1. Schematic of $Hg(NO_3)_2$-$HNO_3$ iodine scrub system and iodate conversion system.

precipitation. A minimum of 2 h was allowed for completion of this step, after which the supernate was returned to the iodine scrub reservoir and the mercuric iodate was retained in the reaction vessel. The entire procedure was repeated until several grams of mercuric iodate had accumulated. The scrub solution capacity of the system was sufficient to allow this type of operation.

The accumulated mercuric iodate was reacted with 1 M sodium hydroxide solution at about 85°C to convert the mercuric iodate to sodium iodate and mercuric oxide. A period of several minutes was allowed for the reaction to go to completion. The resulting mercuric oxide was removed by filtration, reacted with a portion of the scrub solution, then returned to the scrub solution reservoir.

The sodium iodate solution was reacted with barium hydroxide slurry at 85°C to form barium iodate and sodium hydroxide. The barium hydroxide used in this reaction was about 2 M and added in small aliquots, stirring after each addition until precipitation was near completion as determined by spectrophotometric analysis of the supernate for iodate concentration. After initial precipitation of the barium iodate, the supernate was allowed to stand and cool for 1 h to allow more complete precipitation prior to filtration. The sodium hydroxide solution was returned to a reservoir for use in the next cycle. The barium iodate was removed from the filter, dried, and stored. In an actual process, it would be immediately incorporated into concrete for permanent storage.[10,11]

## 3. RESULTS AND DISCUSSION

Two series of experiments were made. The first was conducted to measure the impurities in the barium iodate product when complete solution recycle was employed and to determine if the contamination was detrimental to the final storage of radioactive iodine in cementitious materials. The second was conducted to examine the effects of cross-contamination on the process.

## 3.1 Product Contamination

The most likely contaminants of the barium iodate produced in a scrub and iodate conversion system are mercury and sodium hydroxide. High mercury contamination would be undesirable because it would be a source of heavy-metal poisoning if released to the biosphere. A high concentration of sodium hydroxide could have undesirable effects on the physical characteristics of the concrete or other cementitious materials in which the radioactive iodate is encapsulated.

To determine the type and level of impurities in the final product, five batches of barium iodate were prepared using the bench model and the conditions described previously. The scrub section was run continuously during the preparation period of each batch of barium iodate. At intervals, an aliquot of scrub solution was removed for precipitation and conversion. Total recycle was used in the series. Spectrochemical analyses showed mercury and sodium were present with maximum concentrations of 0.5 and 0.1 wt %, respectively (Table 1). All other contaminants were present at concentrations of $\leq$ 0.5 wt %. The color of the sodium hydroxide solution indicated that it contained mercuric oxide after processing the first batch of barium iodate. However, analytical results showed that it remained in solution and was not extensively occluded or adsorbed in subsequent barium iodate precipitations.

Table 1. Contamination level in barium iodate produced by scrub and conversion system

| Batch No. | Barium iodate (g) | Contamination levels<br>Mercury (wt %) | Sodium (wt %) |
|---|---|---|---|
| 1 | 17.03 | 0.1 | 0.01 |
| 2 | 17.71 | 0.05 | 0.1 |
| 3A | 7.04 | 0.2 | 0.05 |
| 3B | 6.78 | 0.5 | 0.01 |
| 4 | 22.84 | 0.2 | 0.01 |

The low yields of batch 3A and 3B seemed to indicate that the process was not as efficient as desirable. However, it was found that mercuric iodate was caking and accumulating in the evaporator vessel instead of dropping with the solution to the settling-reactor. In a pilot plant or plant installation, provisions would have to be made to prevent such caking and accumulation. The caked material in the evaporator was loosened after batch 3B, and included in the batch 4 product.

A material balance analysis was made after the conclusion of the series to check the efficiency of the system. The distribution of the iodine fed to the system was as follows:

| | |
|---|---|
| Barium iodate product | - 86.74% |
| In-process solutions | - 5.98% |
| Equipment holdup solids | - 4.01% |
| Lost in column off-gas, analytical errors, etc. | - 3.27% |

The iodine compounds held up in the processing solutions should be recoverable with continued operation of the system. The iodine compounds adhering to the system components may represent a steady-state inventory or may continue to build up with system use; this facet of the operation was not investigated. The loss of iodine in the off-gas from the scrub column varies with operating characteristics and would require retrieval by a second iodine scrub system or by solid sorbents. No special effort was made to operate the iodine scrub column for high efficiency because the main interest was to generate material to check for harmful contaminants in the product.

### 3.2 Effect of Cross-Contamination of Process Solutions

A characteristic of any system in which various solutions are recycled is that there may be some unavoidable cross-contamination. In the mercuric nitrate—nitric acid—iodine scrub system combined with a mercuric iodate—to—barium iodate conversion system, the opportunity for cross-contamination exists in the reaction vessel. Here, the mercuric iodate contains a residual amount of mercuric nitrate—nitric acid scrub solution adhering to the precipitated particles. Also, the mercuric oxide formed will contain a residue of sodium hydroxide after the mercuric iodate—to—sodium iodate conversions. Following recycle, if excess barium hydroxide is used in the sodium iodate—to—barium iodate conversion, the sodium hydroxide will contain residual barium hydroxide. This particular contamination is the one most likely to occur and may be detrimental to the operational efficiency. The following reaction would take place if the mercuric iodate was reacted with sodium hydroxide contaminated with barium hydroxide:

$$Hg(IO_3)_2 + 2\ NaOH + Ba(OH)_2 \rightarrow 2\ NaIO_3 + Ba(IO_3)_2 + HgO + H_2O.$$

The barium iodate formed would precipitate and remain on the filter with the mercuric oxide. Then, when the mercuric oxide has reacted with nitric acid to yield the mercuric nitrate—nitric acid solution the barium iodate, being soluble in nitric acid, would go into the column scrub solution.

A series of experiments was conducted to determine the effects of sodium hydroxide containing different amounts of barium hydroxide (Table 2). The percentage of iodine precipitated as $Ba(IO_3)_2$ decreased sharply as the contamination rose to levels greater than a $Ba(OH)_2$:NaOH mole ratio of 1:1100. Iodine remained in the NaOH solution, in the acid scrub solution, and as unreacted $Hg(IO_3)_2$. The latter should not be a problem because it should react in the next cycle. In these tests, 5-g portions of $Hg(IO_3)_2$ were prepared, dried, and ground to a homogeneous state. These samples were washed with 250 ml of fresh iodine scrub column solution, filtered, and then reacted with contaminated sodium hydroxide solutions. The usual procedure was followed for the remainder of the conversion process.

## 4. CONCLUSIONS

Results from a bench-scale mercuric nitrate—nitric acid scrubbing system for gaseous iodine, when combined with conversion of mercuric iodate to barium iodate, showed that the barium iodate produced was suitable for permanent storage in a Portland cement concrete. The

Table 2. Iodine distribution in conversion of mercuric iodate to barium iodate, using 1 M sodium hydroxide contaminated with barium hydroxide

| $Ba(OH)_2$ conc. $\left(\frac{\text{mole } Ba(OH)_2}{\text{moles NaOH}}\right)$ | Unreacted[a] $Hg(IO_3)_2$ | Iodate[b] converted to $Ba(IO_3)_2$ | Unprecipitated[b] iodine compounds | Soluble[c] iodine compounds | Compounds precipitated after filtration |
|---|---|---|---|---|---|
| 0 | | 95.4 | 4.14 | 0.49 | - |
| 1:1100 | 0.07 | 95.4 | 1.90 | 1.46 | - |
| 1:555 | 1.81 | 85.5 | 12.36 | 0.16 | - |
| 1:370 | 0.03 | 86.6 | 9.35 | 3.70 | - |
| 1:275 | 4.17 | 83.1 | 7.27 | 2.03 | 3.52 |
| 1:225 | 10.02 | 51.7 | 30.36 | 1.40 | 6.56 |

Reaction steps:

[a] $Hg(IO_3)_2 + 2\ NaOH \longrightarrow HgO + 2\ NaIO_3 + H_2O$

[b] $2\ NaIO_3 + Ba(OH)_2 \longrightarrow 2\ NaOH + Ba(IO_3)_2$

[c] $HgO + 2\ HNO_3 \longrightarrow Hg(NO_3)_2 + H_2O$

mercury contamination level in the barium iodate produced by the system did not exceed 0.5 wt %, and there was no other element or compound present in sufficient concentration to have an appreciable effect on the storage matrix.

Barium hydroxide contamination of the recycled sodium hydroxide solution used to convert mercuric iodate to sodium iodate was the major deterrent to efficient operation of the combined system. A $Ba(OH)_2$: NaOH mole ratio of 1:225 decreased the final yield of barium iodate product to 51.7%. Strict control would be required in the amount of barium hydroxide used in the conversion of sodium iodate to barium iodate to minimize cross-contamination. The iodate content could be determined before the conversion so that less than the stoichiometric amount of barium hydroxide could be used. The unreacted sodium iodate would be recycled with the sodium hydroxide and would cause no adverse effects if controlled at low concentrations. This technique, combined with sufficient reaction time, would ensure that barium hydroxide would not accumulate in the sodium hydroxide solution.

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