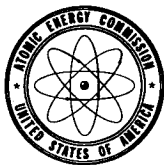


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AEC-NASA TECH BRIEF



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Synthesis of Perbromates

The problem:

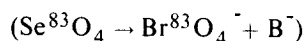
To prepare perbromates. Past attempts to prepare perbromates—salts of heptavalent bromine—were generally unsuccessful. Early reports of such compounds cannot be confirmed.

The solution:

Successful synthesis of perbromates and isolation of the rubidium salt by a hot-atom process.

How it's done:

Perbromate was synthesized by a hot-atom process—the beta decay of radioactive Se^{83} incorporated into a selenate:



Enriched Se^{83} (90%) was irradiated with thermal neutrons, dissolved in nitric acid, and oxidized to selenate by ozone in alkaline solution. After decay of the 25-min Se^{83} activity sodium perchlorate and bromate were added, and rubidium perchlorate was precipitated at 0°C under conditions such that only 1% of the bromate was coprecipitated. Approximately 14% of the 2.4-hr Br^{83} activity was found in the precipitate. After treatment with iodide in 6M HCl, however, only about 1% of the Br^{83} activity coprecipitated with rubidium perchlorate.

These results indicated the formation of a relatively unreactive perbromate ion and led to the preparation of macro amounts of perbromates.

An electrolytic cell was set up having a platinum cathode immersed in 3 ml of 2.8M HClO_4 in a porous-porcelain cup. The anode was a rotating platinum microelectrode immersed in a slurry of Li_2CO_3 in 3

ml of 2.8M LiBrO_3 tagged with 36-hr Br^{82} in a similar cup. The two cups were placed in a container of 2.8M LiClO_4 , which in turn was immersed in a cooling bath at -15°C. The cell was run for about 1 A-hr at an anodic-current density of about 20 A/cm². Successive portions of Li_2CO_3 were added to the anolyte to maintain its neutrality, while the catholyte was periodically replaced for maintenance of its acidity.

At the end of the electrolysis, about 2% of the bromine activity coprecipitated with RbClO_4 , but not with $\text{Ba}(\text{BrO}_3)_2$. When electrolysis was carried out at a similar density but in an unpartitioned cell with dichromate used to inhibit cathodic reduction, essentially all the bromide activity could be coprecipitated with $\text{Ba}(\text{BrO}_3)_2$. A technique was also developed for analysis for perbromate in the presence of bromate.

In connection with this study, perbromic acid was shown to be a stable compound, and its mass spectrum was determined; spectra from a heated sample persisted for 1 hr. The volatility of perbromic acid lies between those of perchloric acid and periodic acid.

Notes:

1. Inquiries concerning this innovation may be directed to:

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(continued overleaf)

Patent status:

Inquiries concerning rights for commercial use of this innovation may be made to:

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