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Applying new ideas for the distillation of osmium

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A very efficient, rapid, low blank method for the extraction of Osmium from geological materials will be presented. As with existing techniques, this new method relies upon the oxidation of Os in the sample solution to OsO_4 and utilises the volatile nature of this oxide. In contrast to previous distillation methods where an Os oxide-bearing carrier gas is bubbled through chilled HCl or HBr, however, the Os is caught in sulfuric acid coated on the inner surface of a small-scale cold trap. Once the distillation is completed, the trapped Os is extracted into 500 µl HBr conc. and can be subjected to a final cleaning step employing conventional micro-distillation techniques [1].

The entire extraction procedure takes app. 60 min. at 65 °C and requires only 2 µl H_2SO_4 to trap the osmium, which is achieved with great efficiency. Thus, apart from the digestion medium, the extraction and recovery of Os using this technique employs a total of only 502 µl of reagents instead of 5 - 10 ml used in conventional procedures. The efficiency of the method and the low reagent volumes required for the distillation produce yields of more than 80% and total procedural blanks of only 50 - 100 fg Os on a routine basis.

The method is well suited to perform highly precise Re-Os analyses on various sample types, such as whole rocks (OFB, OIB & IAV), soils, coal, sulphides, limestones and phosphates (conodonts).

References

[1] Naegler Th. F. & Frei R. (1997): Plug in osmium distillation. *Sch. Mineral. Petrogr. Mitt.* **77**, 123-127.

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Influence of residue after HF-HClO$_4$ digestion on bulk REE determination

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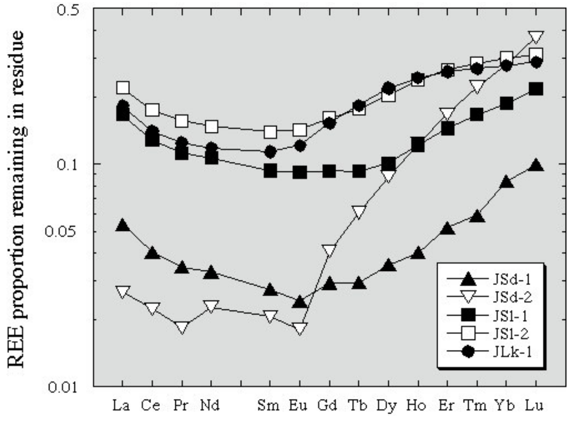
In order to estimate the influence of residue remaining after acid digestion on bulk REE determination, we analyzed REEs in basaltic and sedimentary rocks and synthesized fluorides using methods employing both acid (HF-$HClO_4$) digestion and alkali (Na_2CO_3-H_3BO_4) fusion. REE analyses of the standard sample BCR-1 show good reproducibility and close agreement with compilation values, indicating the accuracy of the present analytical method.

Analyses of ralstonite formed from JB-1a and AlF_3 formed from JSd-1 indicate that ralstonite preferentially incorporates HREE compared to LREE and that AlF_3 has extreme LREE enrichment. However, the mineralogy of secondary fluorite depends on the digested sample size as well as the chemical composition of the sample. It is difficult, therefore, to predict which fluoride will be precipitated during digestion of a certain amount of a sample. Secondary precipitates have a significant influence on bulk REE determinations of sedimentary rocks: up to 30% Lu can be incorporated into secondary fluorides, even if the digested sample size is less than 100mg.

Smooth REE patterns of sedimentary reference rocks suggest that the combined method of acid digestion and alkali fusion is an effective method for recovering REEs in sedimentary rocks for bulk REE determination.



Plot of REE proportion remaining in the digestion residues against total REE of respective rock samples (JSd-1, -2, JSl-1, -2 and JLk-1). The pattern of JSd-2 suggests the existence of an undigested mineral such as zircon.