New methods for iron isotope measurements in ancient sedimentary rocks

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- **Fe isotopes are likely to be an important tracer for finding traces of possible life during future missions to other planets in our solar system**
- Current Mullticollector ICP-MS-based measurements techniques are prone to substantial bias due to inadequate sample preparation.
- Fe isotopes vary over a limited range in nature (order of 3.5‰), and highly precise and accurate isotopic measurements are thus essential
- Here we aim to obtain a 100% Fe yield after column extractions and completely remove all possible interfering elements to give accurate results

MATERIALS & METHODS:

- **We explored a range of previously published techniques (Dauphas et al. 2009, Dauphas and Rouxel, 2006 and Schoenberg and von Blanckenburg, 2005) for Fe extraction from Fe-rich rocks including banded iron formations, shales and pyrite**

✧ Two different resin types were tested:
AG1-X8 200-400 and AG1-X8 100-200 mesh Cl^- form anion exchange resin.

Artificial solution
10 ppm Fe
2ppm Ca
2ppm Cr
2ppm Ni
2ppm Ti

Rock reference material
BHVO-2 (basalt) → 8.63% Fe
MAG-1 (fine grained clayey mud) → 4.75 wt% Fe
ECSC 681-1 (iron ore) → 33.21wt % Fe

Mineral sample
Pyrite
Particle dimension: 60-120 µm
~ 45 wt% Fe

✧ Two different oxidizing agents (H_20_2 and $HClO_4$) were used to keep Fe in its Fe^{3+} form in order to obtain a 100% recovery of iron.

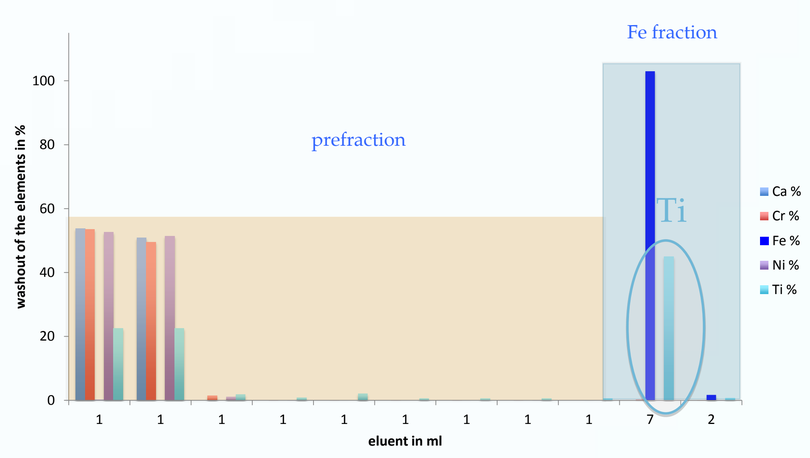
Iron isotpes will be measured on materials obained from a International Continental Drilling Program (ICDP) in Barbeton Greenstone Belt in South Africa

RESULTS:

Four different methods were performed to test 100% recovery of iron:

1. Elution with 6.0M HCl:
   ~2% Fe lost in prefraction
2. Elution as in 1) with 6.0M HCl +0.001% H_2O_2
3. Elution as above but with different resin particle size
4. Elution of the prefraction was performed testing different chemical (6.0M HCl + 0.01M $HClO_4$)

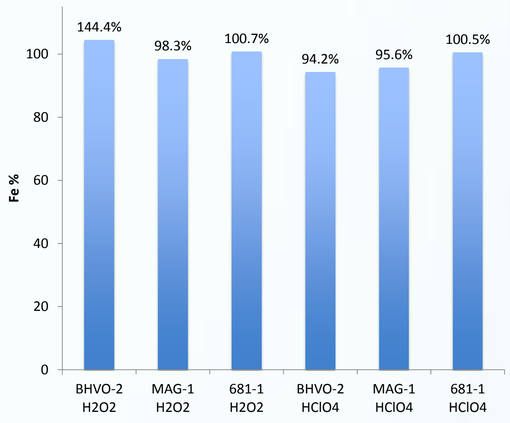
Elution with perclorate; Ti follows Fe:



Reference material test:

Two different elution techniques were tested and two column extractions performed:

1. HCl + 0.001%H_2O_2
2. HCl+ 0.01M $HClO_4$

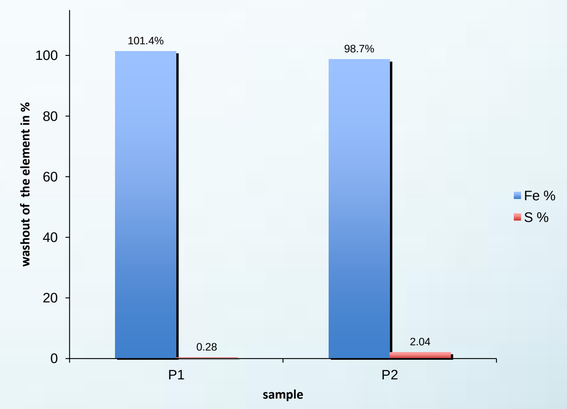


Fe yield ~100%
Ti is completely eliminated after the 2nd column extraction

Pyrite test:

Pyrite samples are dissolved in:

•10 drops HF conc., 6 drops HNO3 conc. and 1 drop HClO4 conc.
•Prefraction eluted with 6.0M HCl+0.01M $HClO_4$
•Fe fraction eluted with 0.4 M HCl



Fe yield ~ 100%, no S

CONCLUSIONS:

- **~ 100%Fe yield in all tests**
- **HCl only → risk of losing some Fe in prefraction**
- **$HClO_4$ and H_2O_2 agents result both in 100% yields. However $HClO_4$ is preferred because it is more stable during the evaporation of solutions in subsequent steps of the method.**
- **Ti follows Fe chemistry but gets eliminated in 2nd extraction**
- **The pyrite test resulted in good yields without sulpher in the final iron solution**
- **The collection of the Fe from the columns can be done without $HClO_4$ for pyrite samples**

References cited:
Dauphas et al., Chemical Geology 267 (2009), **Dauphas and Rouxel**, Mass Spectrometry Reviews 25 (2006), **Schoenberg and von Blanckenburg,** International Journal of Mass Spectrometry 25 (2005)