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**Supporting information for article:**

### **Iterative energy self-calibration of Fe XANES spectra**

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**S1. Methods:**

*Samples:* The composition of the samples is given in the Table S1.

**Table S1** Major element composition in weight percent of studied samples

	Basaltic glass standards <sup>1</sup>	MORB glass VG 3450 <sup>1</sup>	Rhyolite glass standards <sup>2</sup>	RGM-2 <sup>3</sup>	Havre rhyolitic pumice
SiO <sub>2</sub>	52.10 (0.40)		73.85 (2.10)	73.4 (0.41)	73.1 (1.28)
Al <sub>2</sub> O <sub>3</sub>	15.87 (0.22)		10.44 (1.00)	14.0 (0.30)	13.49 (0.88)
CaO	12.33 (0.17)		0.24 (0.02)	1.23 (0.03)	2.35 (0.42)
FeO <sup>tot</sup>	9.40 (0.22)	11.87	4.85 (1.69)	1.86 (0.04)	2.34 (0.21)
MgO	7.77 (0.09)	4.56	0.04 (0.03)	0.28 (0.02)	0.42 (0.06)
TiO <sub>2</sub>	1.23 (0.03)		0.24 (0.02)	0.25 (0.02)	0.37 (0.04)

<sup>1</sup> (Berry *et al.*, 2018)

<sup>2</sup> (Cottrell *et al.*, 2009)

<sup>3</sup> United States Geological Survey: Rhyolite, Glass Mountain 2

*XANES energies:* The incident X-ray energy spacing used is detailed in Table S2.

**Table S2** XANES energy spacing

Energy Range (eV)	Energy Spacing (eV)
6962 to 7102	10
7102 to 7122	0.25
7122 to 7162	0.5
7162 to 7262	4
7262 to 7462	10

**S2. Matlab® Code:**

The following code was written in Matlab®, but can easily be translated to other languages.

```
%%%%%%%%%%%%%%%
%%%%%
% Self-calibration of Fe k-edge XANES spectra
% Michael Jones. Copyright (c) 2019
% Queensland University of Technology
% mw.jones@qut.edu.au
% Uses "crossing.m" Copyright (c) 2016, Steffen Brueckner
% https://au.mathworks.com/matlabcentral/fileexchange/2432-crossing
% All energy values in keV unless otherwise stated
%%%%%
%%%%%
FeDataNorm; %vector with normalised Fe spectra
Energies; %vector with energies corresponding to FeDataNorm
A_Fit; %linear fit to the standards using Method A
B_Fit; %linear fit to the standards using Method B
Fe3Full; %vector of Fe3+ concentration values from 0 to 1 corresponding to the linear fits (A_Fit and B_Fit)
Yint=ones(size(Energies))*0.8; %define point where the edge intensity will be measured
Fe3percentA=1; %Fe3+ concentration using Method A: starting value
Fe3percentB=0; %Fe3+ concentration using Method B: starting value
EDiff=1; %Difference between the energy reported by Method A
%and the energy required for the concentration reported by the
%mean Methods A and B: starting value
EnergyDiff=[]; %Empty array for storing convergence data

while abs(EDiff)>1e-7 %EDiff is in keV

    %Get the 0.8 crossing point and find the Fe3 at that point
    [~,EnergyX]=crossing(YDataNorm,Energies,0.8);
    [~,Fe3percentA]=crossing(A_Fit,Fe3Full,EnergyX(end));

    %get the two post edge points at energies E1 and E2 and get the Fe3 at that point
    [~,I1]=crossing(Energies,YDataNorm,E1);
    [~,I2]=crossing(Energies,YDataNorm,E2);
    RatioI=I2/I1;
    [~,Fe3percentB]=crossing(B_Fit,Fe3Full,RatioI);
```

```
if isempty(Fe3percentA)|| isempty(Fe3percentB)
%If the Fe3 concentration can't be found via either method, throw an error: You may need to shift the spectra
to within range.
error('Fe3+ could not be determined: Is the unknown the same type as the standards or do you need to
manually shift the spectra within range?')
end

Fe3Mean=(Fe3percentA+Fe3percentB)/2; %The mean of Fe3+ from Method A and B
[~,Energy2]=crossing(Fe3Full,A_Fit,Fe3Mean); %The edge energy where a sample with Fe3+ = Fe3Mean
would be expected
EDiff=Energy2-EnergyX; %The difference in the energy between Method A and the mean of A and B
Energies=Energies+EDiff; %Update the energy
EnergyDiff=[EnergyDiff; [EDiff Fe3percentA Fe3percentB]]; %Store the convergence data

end

dlmwrite('CalibratedEnergy.txt',Energies); %Write out the self-calibrated energy list
```

### S3. References

- Berry, A. J., Stewart, G. A., O'Neill, H., St.C., Mallmann, G. & Mosselmans, J. F. W. (2018). *Earth and Planetary Science Letters* **483**, 114-123.
- Cottrell, E., Kelley, K. A., Lanzirotti, A. & Fischer, R. A. (2009). *Chemical Geology* **268**, 167-179.