Rapid, Accurate Metallic Iron Analysis for Ironmaking Materials

Daniel J. C. Stewart Energy Safety Research Institute, Swansea University D.J.C.Stewart@Swansea.ac.uk

Why is Fe^0 analysis critical for Ironmaking?

- Strong bearing on the **value in use** of recycled materials like slags and revert dusts.
- Used to measure the quality of **Direct Reduced Iron (DRI).**
- Using metallized raw materials **decreases reductant rates** and **CO_2 emissions.**

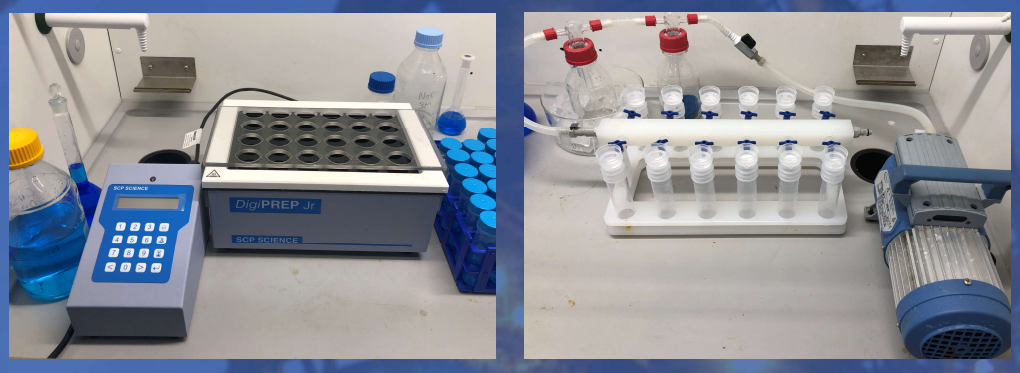
Existing Protocol

- ISO 5416:2006 details methanol bromide titrimetric method, **slow and laborious.**
- $CuSO_4$ for selective dissolution on hotplate followed by hot filtration, dilution and **ICP-MS** is industry standard.
  - Inconsistent heating
  - Cannot run unattended.
  - **Manual stirring is required.**
  - Large volumes of ecotoxic aqueous waste (250ml per sample).
  - Low sample throughput – **4 samples p/h.**
  - **±1.0% accuracy.**

Current testing protocol for metallic Fe is **slow, labour intensive** and **cannot be run unattended**

New Protocol using DigiPREP heating block

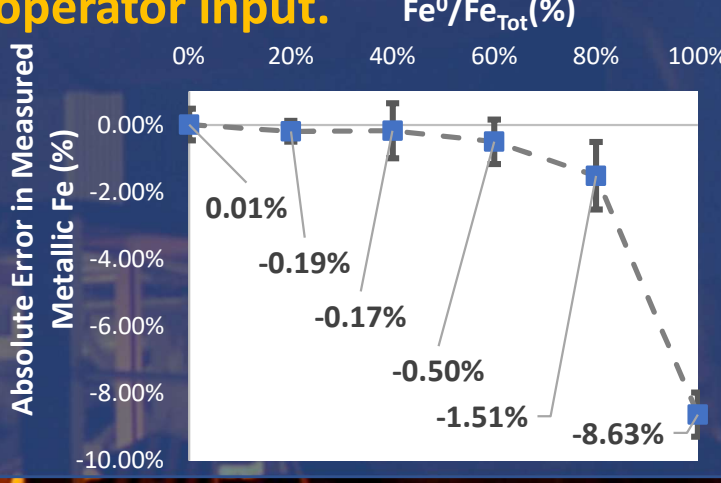
- ≈0.25g <65 µm analyte powder in 20 cm^3 in 0.25 M $CuSO_4$ heated for 90 mins at 100 °C.
- Hot vacuum filtered using 12 position manifold and acidified with HCl (37%, 3 cm^3) and made to 50 cm^3 volume within reaction tubes and diluted for ICP-MS analysis.

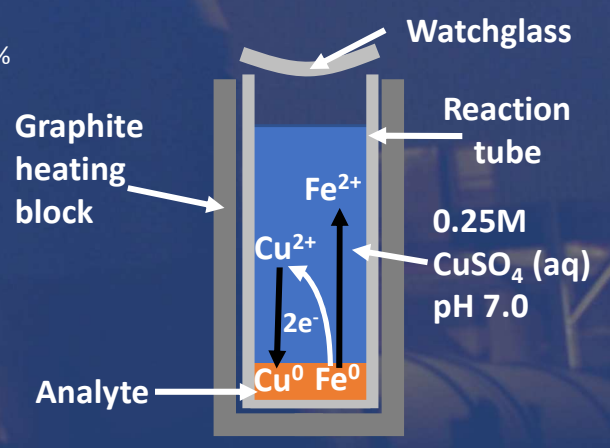


Heating block and controller (Left) and hot vacuum filtration apparatus (Right).

DigiPREP Analysis Validation and Conclusions

- Random error = **±0.74%.**
- Determinate error increases with increasing Fe^0.
- Analysis has been validated to be accurate **within the margin of random error up to 150mg Fe^0.**
- Current $CuSO_4$ concentration appears **insufficient to oxidize** all metallic Fe present. **Further optimization is required.**
- **18 samples per hour can be produced with minutes of operator input.**





New analysis has comparable **±1.0%** accuracy but **more productive**, **less operator time** and with **less waste**