Rapid, Accurate Metallic Iron Analysis for Ironmaking Materials

Why is Fe₀ 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 CO₂ emissions.

Existing Protocol
• ISO 5416:2006 details methanol bromide titrimetric method, slow and laborious.
• CuSO₄ 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 cm³ in 0.25 M CuSO₄ heated for 90 mins at 100 °C.
• Hot vacuum filtered using 12 position manifold and acidified with HCl (37%, 3 cm³) and made to 50 cm³ volume within reaction tubes and diluted for ICP-MS analysis.

DigiPREP Analysis Validation and Conclusions
• Random error = ±0.74%.
• Determinate error increases with increasing Fe₀.
• Analysis has been validated to be accurate within the margin of random error up to 150mg Fe₀.
• Current CuSO₄ 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.