

	Geochemistry Lakefield Laboratory	Doc Type Method Summary Method Code GC_CLA01V Service Testing Issued Date April 2022 Approved by V. Bautista
	Natural Resources	Ferrous Iron Determination by Titration with Potassium Dichromate

1. Parameter(s) measured, unit(s):

Fe²⁺, in %

2. Typical sample size:

0.25g

3. Type of sample applicable (media):

Rocks and ores, ferrous concentrates (also iron ore pellets, direct reduced iron, hot-briquetted iron) and other iron-bearing materials (slag, mill scale).

This test method will not perform effectively on samples that contain organics. Metallic iron in significant amounts may interfere.

4. Sample preparation technique used:

The sample is digested with sulphuric and hydrofluoric acid at high temperature. Any HF left is converted into fluoroboric acid by the addition of boric acid immediately after the acid digestion.

5. Method of analysis used:

The sample is quickly cooled and the ferrous iron is titrated with a standardized solution of potassium dichromate resulting in the oxidation of the ferrous (Fe⁺²) ion to the ferric (Fe⁺³) ion. Endpoint is detected visually using barium diphenylamine sulfonate as the external indicator. Fe²⁺ as FeO is reported where the raw data as Fe²⁺ is multiplied by 1.286.

6. Data reduction by:

Manual data entry of the titration volume into the worksheet, data fed to Laboratory Information Management System with secure audit trail.

7. Figures of Merit:

This method has been fully validated for the range of samples typically analyzed. Method validation includes the use of reference materials, replicates, duplicates and blanks to calculate accuracy, precision, linearity, range, limit of detection, reporting limit, specificity and measurement uncertainty.

The Reporting Limit has been determined according to the following:

Element	Fe²⁺
RL(%)	0.5
Upper limit (%)	100

8. Quality control:

Quality control materials include method blanks, replicates and reference materials and are randomly inserted with the frequency set according to method protocols at 18% for process control analysis. Quality control materials will also include BRM (Barren reference materials, or preparations blanks) and preparation duplicates if samples have been taken through the sample reduction process. Standardization of titrant performed per bottle.

9. Accreditation:

SGS Natural Resources conforms to the requirements of ISO/IEC 17025. Scopes of Accredited tests are site specific, please visit <https://www.scc.ca/en/search/laboratories>