

Code 4F

Analysis	Method	Detection Limit	Upper Limit
FeO	Titration	0.01%	-
S	Infrared	0.01%	40%
SO ₄	Infrared	0.3%	-
SO ₃	Infrared, Calc.	0.3%	-
Sulphide	Infrared, Calc.	0.3%, 0.01%	-
Cl	INAA	0.01%	-
Cl (ISE)	FUS-ISE	0.01%	-
Hg	Cold Vapour FIMS	5 ppb	100,000 ppb
F	FUS-ISE	0.01%	-
CO ₂	Infrared	0.01%	-
Total H	Infrared, Calc.	0.01%	-
H ₂ O +/-	Infrared	0.1%	-
Total H ₂ O	Infrared	0.1%	-
C, S	Infrared	0.01%	-
B	PGNAA	0.5 ppm	10,000 ppm
B	PGNAA	0.2 ppm	10,000 ppm
Total N	Thermal Conductivity	0.01%	-
CaCO ₃	Infrared, Calc.	0.02%	-

Code 4F - FeO

FeO is determined through titration, using a cold acid digestion of ammonium metavanadate, and hydrofluoric acid in an open system. Ferrous ammonium sulphate is added after digestion and potassium dichromate is the titrating agent.

This cold digestion will dissolve silicates and some sulphides. Pyrite may not be totally dissolved. The extent of dissolution is affected by the ferric iron content higher Fe(3+) concentration increases the solubility of pyrite. Concentrations of S(II) has a tendency to reduce Fe(3+) to Fe(2+). This is minimized in an open system. If the S concentration is greater than 10% an alternative hot digestion method is used for FeO.

When titrating, the endpoint is determined by colour. Certain solutions high in MnO₂ or certain matrices make the endpoint determination difficult or impossible.

Modified method from Wilson (1955. Bull Geol. Sur. Gt. Britain, volume 9, pp. 56-68).

Code 4F - S

Accelerator material is added to a 0.2 g sample. The inductive elements of the sample and accelerator couple with the high frequency field of the induction furnace. The pure oxygen environment and the heat generated by this coupling cause the sample to combust. During combustion, sulphur-bearing elements are reduced, releasing sulphur, which binds with oxygen to form SO₂. Sulphur is measured as sulphur dioxide in the infrared cell. An Eltra CS-2000 or a Leco CNS 2000 is used for analysis.

Code 4F - SO₄

A 0.2 g sample is combusted in a resistance furnace at 550 °C in a pure oxygen environment. During combustion, sulphur-bearing elements are reduced, releasing sulphur, which binds with oxygen to form SO₂. Sulphur is measured as sulphur dioxide in the infrared cell. An Eltra CS-2000 or a Leco CNS 2000 is used for analysis.

Code 4F - SO₃

Sulphite sulphur is calculated from the sulphate sulphur.

Code 4F - Sulphide

Total sulphur is determined on the sample at 1450 °C following the measurement at 550 °C. Sulphide sulphur is determined as the difference between total sulphur and sulphate sulphur.

Code 4F - Hg

0.5 g sample is digested with aqua regia to leach out soluble compounds. The Hg in the resulting solution is oxidized to the stable divalent form. An aliquot of the digestion solution is mixed with a potassium permanganate solution. Using the flow injection cold vapor mercury technique, Hg (II) is reduced to mercury vapour using stannous chloride, argon is bubbled through the mixture of sample, carrier, and reductant solutions in a closed reaction system to liberate and transport the Hg atoms into an absorption cell. Hg is determined via the absorption of light at 253.7 nm. The maximum amount absorbed (peak height) is directly proportional to the concentration of mercury atoms in the light path. The sensitivity of the cold vapor technique is far greater than can be achieved by conventional flame AA. A Perkins Elmer FIMS 100 is used for the analysis.

Code 4F - Cl

1 g samples are weighed into medium poly vials and are irradiated at a thermal flux of $7 \times 10^{12} \text{ n cm}^{-2} \text{ s}^{-1}$ in a computer controlled rabbit system. After appropriate decay times to allow aluminum and other very short lived isotopes to decay, the samples are counted sequentially for analyte(s) of interest. Values are corrected for decay and compared to a standard calibration. Two standards are analyzed with every work order. Duplicates are analyzed when samples are provided.

Code 4F - Cl (ISE)

0.2 g samples are fused with a combination of lithium metaborate and lithium tetraborate in an induction furnace to release the chloride ions from the sample matrix. The fuseate is dissolved in dilute nitric acid, prior to analysis the ion strength of the solution is adjusted. The chloride ion electrode is immersed in this solution to measure the fluoride-ion activity directly.

Code 4F - F

0.2 g samples are fused with a combination of lithium metaborate and lithium tetraborate in an induction furnace to release the fluoride ions from the sample matrix. The fuseate is dissolved in dilute nitric acid, prior to analysis the solution is complexed and the ionic strength adjusted with an ammonium citrate buffer. The fluoride ion electrode is immersed in this solution to measure the fluoride-ion activity directly. An automated fluoride analyzer from Mandel Scientific is used for the analysis.

Code 4F - CO₂

0.2 g sample is thermally decomposed in a resistance furnace in a pure nitrogen environment at 1000 °C, using an ELTRA CW-800, directly releasing CO₂. H₂O is removed in a moisture trap prior to the detection of carbon dioxide in the IR cell. Carbon dioxide absorbs IR energy at a precise wavelength within the IR spectrum. Energy from the IR source is absorbed as the gas passes through the cell, preventing it from reaching the IR detector. All other IR energy is prevented from reaching the IR detector by a narrow bandpass filter. Because of the filter, the absorption of IR energy can be attributed only to carbon dioxide (CO₂). The concentration of CO₂ is detected as a reduction in the level of energy at the detector.

Code 4F - Total H

0.3 g sample is thermally decomposed in a resistance furnace in a pure nitrogen environment at 1000 °C, using an ELTRA CW-800, directly releasing H₂O, which includes both H₂O⁻ and H₂O⁺. H₂O absorbs IR energy at a precise wavelength within the IR spectrum. Energy from the IR source is absorbed as the gas passes through the cell, preventing it from reaching the IR detector. All other IR energy is prevented from reaching the IR detector by a narrow bandpass filter. Because of the filter, the absorption of IR energy can be attributed only to water (H₂O). The concentration of H₂O is detected as a reduction in the level of energy at the detector. Total H is calculated from H₂O.

Code 4F - H₂O +/-

0.3 g sample is thermally decomposed in a resistance furnace in a pure nitrogen environment at 110 °C (moisture, H₂O⁻) followed by decomposition at 1000 °C (interstitial water, H₂O⁺), using an ELTRA CW-800, directly releasing H₂O. H₂O absorbs IR energy at a precise wavelength within the IR spectrum. Energy from the IR source is absorbed as the gas passes through the cell, preventing it from reaching the IR detector. All other IR energy is prevented from reaching the IR detector by a narrow bandpass filter. Because of the filter, the absorption of IR energy can be attributed only to water (H₂O). The concentration of H₂O is detected as a reduction in the level of energy at the detector.

Code 4F - Total H₂O

0.3 g sample is thermally decomposed in a resistance furnace in a pure nitrogen environment at 1000 °C, using an ELTRA CW-800, directly releasing H₂O, which includes both H₂O⁻ and H₂O⁺. H₂O absorbs IR energy at a precise wavelength within the IR spectrum. Energy from the IR source is absorbed as the gas passes through the cell, preventing it from reaching the IR detector. All other IR energy is prevented from reaching the IR detector by a narrow bandpass filter. Because of the filter, the absorption of IR energy can be attributed only to water (H₂O). The concentration of H₂O is detected as a reduction in the level of energy at the detector.

Code 4F - C, S

Accelerator material is added to a 0.2 g sample. The inductive elements of the sample and accelerator couple with the high frequency field of the induction furnace. The pure oxygen environment and the heat generated by this coupling cause the sample to combust. During combustion, carbon-bearing elements are reduced, releasing the carbon, which immediately binds with the oxygen to form CO and CO₂, the majority being CO₂. Also, sulphur-bearing elements are reduced, releasing sulphur, which binds with oxygen to form SO₂. Sulphur is measured as sulphur dioxide in the first IR cell. A small amount of carbon monoxide is converted to carbon dioxide in the catalytic heater assembly; SO₂ is converted to SO₃, while sulphur trioxide is removed from the system in the cellulose filter. Carbon is measured as carbon dioxide in the IR cell as gases flow through the IR cells. Carbon dioxide absorbs IR energy at a precise wavelength within the IR spectrum. Energy from the IR source is absorbed as the gas passes through the cell, preventing it from reaching the IR detector. All other IR energy is prevented from reaching the IR detector by a narrow bandpass filter. Because of the filter, the absorption of IR energy can be attributed only to carbon dioxide (CO₂). The concentration of CO₂ is detected as a reduction in the level of energy at the detector. An Eltra CS-2000 is used for the analysis.

Code 4F - B (0.5 ppm and 2 ppm B)

1 g samples are encapsulated in a polyethylene vial and placed in a thermalized beam of neutrons produced from a nuclear reactor. Samples are measured for the doppler broadened prompt gamma ray at 478 KeV using a high purity GE detector. Samples are compared to certified reference materials used to calibrate the system. A minimum of four standards are analyzed with every work order. Duplicates are analyzed when samples are provided. The detection limit reported is a function of the counting times required for each.

Code 4F - Total N

0.2 g sample is combusted in a resistance furnace at 1350 °C, using a LECO CNS-2000. Combustion gases are collected in a 4.5-liter ballast tank and then flow to the detectors. Nitrogen, as N₂ is detected by thermal conductivity detection. A Leco CNS-2000 is used for the analysis.

Code 4F - CaCO₃

0.2 g sample is thermally decomposed in a resistance furnace in a pure nitrogen environment at 1000 °C, using an ELTRA CW-800, directly releasing CO₂. H₂O is removed in a moisture trap prior to the detection of carbon dioxide in the IR cell. Carbon dioxide absorbs IR energy at a precise wavelength within the IR spectrum. Energy from the IR source is absorbed as the gas passes through the cell, preventing it from reaching the IR detector. All other IR energy is prevented from reaching the IR detector by a narrow bandpass filter. Because of the filter, the absorption of IR energy can be attributed only to carbon dioxide (CO₂). The concentration of CO₂ is detected as a reduction in the level of energy at the detector. CaCO₃ is calculated from CO₂.