

IRON ASSAY USING ICP-OES

Put samples in glass vials.

If needed, dry under blowing argon and in 60°C water bath.

Add 1mL of a mixture of 5vols of concentrated nitric acid to 2vols concentrated perchloric acid.

Cap loosely and put on a 100°C heat block in a fume hood.

Allow to digest (wet ash) several hours to overnight.

Make up to 1mL with more nitric/perchloric acid. Mix well.

Transfer 250µL to another tube and add 1mL water for a final volume of 1.25mL.

Measure iron content with a Perkin Elmer 3100XT Inductively Coupled Plasma Optical Spectrometer system (ICP-OES).

As a blank, digest 1.0mL of the nitric/perchloric acid mixture in the same batch of vials together with the samples. Subtract its raw reading from that of the samples. Any difference reading near or below 0.050ppm is not easily reproducible on this instrument.

A standard solution of 0.500ppm of iron was used in the quantitation.