

Persulfate digestion

Principle

Persulfate oxidizes dissolved organic N (DON) to NO_3^- . NO_3^- may then be determined by any one of a number of standard methods. Persulfate oxidation has been developed for soil extract DON (Cabrera and Beare, 1993) and for fresh water DOC (McDowell et al., 1987). It is often used in conjunction with chloroform fumigation to determine microbial N in soils.

Persulfate oxidation depends on peroxydisulfate ($\text{K}_2\text{S}_2\text{O}_8$) decomposition into the persulfate radical (HSO_4^\bullet), which is the active oxidizing agent. This decomposition follows an Arrhenius relationship between 50 and 130°C (Kolthoff and Miller, 1951; Goulden and Anthony, 1978); persulfate has a half-life of about 30 s at 130°C and 4 h at 75°C. The decomposition is the rate-limiting step, and further oxidation steps are rapid relative to free radical initiation (Peyton, 1993). Under some conditions, higher temperature may decrease C recovery (Goulden and Anthony, 1978). Thus, high temperature may increase reaction rate but not necessarily completeness.

Method adapted from:

Allen Doyle,* Michael N. Weintraub, and Joshua P. Schimel (2004) Persulfate Digestion and Simultaneous Colorimetric Analysis of Carbon and Nitrogen in Soil Extracts. *Soil Sci. Soc. Am. J.* 68:669–676 (2004).

Prepare stock solutions

Make persulfate reagent

- In a 1000-mL beaker dissolve 50 g of $\text{K}_2\text{S}_2\text{O}_8$, 16.8 g of NaOH, 30g of H_3BO_4
- Transfer to a 1000-mL volumetric flask and make up to 1L
- Solution is stable for up to 8 days in the dark at 4 °C

Make 10 mM amino-N stock solution (for testing recovery)

- Dissolve 0.075 g of glycine in 100 mL of DI water
- Store in dark at 4°C

Make 200 uM Amino-N standard (for testing recovery)

- Pipette 2mL of 10 mM stock solution into 100 mL vol flask
- Make up to 100.0 mL using extractant or sample matrix

Analysis procedure

- Pipette equal amounts of persulfate reagent and sample into a suitable container (e.g. 5mL of each in a 10-mL container)
- Carry at least three 200-uM amino N standards through the same procedure
- Sit the cap on top but don't close it otherwise it may explode
- Place in an oven at 80 °C over night
- After cooling determine $[\text{NO}_3^-]$. Check that recovery of glycine as nitrate is >90%