

# NOSAMS Facility

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METHOD: Acid fumigation of particulate organic carbon samples  
EFFECTIVE DATE: 1/25/2023  
APPROVED BY: Dr. Roberta Hansman

**PURPOSE:** To remove inorganic carbon from particulate organic carbon samples by acid fumigation in preparation for radiocarbon analysis.

**INTRODUCTION:** Particulate organic carbon samples that are received on filters require the removal of inorganic carbon (carbonates) through vapor phase acidification. Additionally, inorganic carbon from some sediment samples may be removed using this method.

## MATERIALS & APPARATUS

- Concentrated HCl (12 M, trace metal grade)
- NaOH pellets
- Glass petri dishes
- Glass vacuum-sealed desiccator(s) with o-ring seals (o-ring must be compatible with HCl and NaOH, e.g., Viton)
- Nitrile gloves and clean utensils (spatula, forceps etc.)
- Oven (65 °C, in fume hood)
- Drying oven (60 °C)

**PROCEDURE:** Weigh or place samples into glass petri dishes and record sample information. Wear gloves and use only clean utensils to handle samples.

*NOTE: Do not label petri dishes with marker, but rather use dishes with etched numbers or letters for identification.*

Place glass vacuum desiccator in an oven (the oven should be in a fume hood).

Add 50 ml concentrated HCl (12 M) in a beaker or petri dish, and place at the bottom of the desiccator.

Place a porcelain (no metal or plastic) desiccator plate above the acid; it should fit snugly in the desiccator groove.

Place samples in petri dishes in the desiccator onto the plate.

Seal and gently evacuate the desiccator.

Turn on the oven, set at 65 °C, and fume samples for 72 h.

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After the allotted time, place samples in another glass vacuum desiccator with a beaker of NaOH pellets (about 50 g), or (if only one desiccator available) switch out the petri dish of HCl for one with NaOH pellets. Use care when breaking the vacuum seal and proceed slowly.

Seal and gently evacuate the desiccator, as before.

Neutralize the samples in the oven at 65 °C for another 72 h.

After neutralization, dry samples in 60 °C drying oven for at least a few hours or overnight.

**RECORDS:** Information specific to the processing of each sample is recorded in a notebook and in the NOSAMS relational database including operator, unique receipt number, mass, pretreatment method, and comments.