STANDARD OPERATING PROCEDURE

SAMPLE PREPARATION AND SUBMISSION FOR TOC ANALYSIS

Introduction and principles of the technique

The Elementar Vario TOC cube uses high-temperature catalytic combustion of up to 1200°C and CO_2-measurement with a nondispersive infrared (NDIR) sensor for detection of Total organic carbon (TOC), Non-purgeable organic carbon (NPOC), Total Carbon (TC), Total Inorganic Carbon (TIC) and Total bound Nitrogen (TNb) is measured simultaneously with an electrochemical detector (ECD).

Types of samples which can be analysed

- **Soils/ Sediments/ Sludge**
- **Liquid** - water, surface and saline waters, and domestic and industrial wastes

Procedure for preparing liquid samples

- Collect at least 50 ml samples in polyethylene bottles/ vials which are free from carbonate/organic contamination
- During sample collection, bottles should be filled to the brim with no head space to avoid CO_2 absorption from the air in aqueous samples.
- Environmental samples should be kept cool (4°C) on ice in the dark to limit the possibility of bacterial decomposition of some components. The samples should be submitted for analysis as soon as possible (TOC analysis of acidified samplescan be completed within 28 days if stored correctly)
- Label samples according to your sample submission template (downloadable on the website).

Sample submission and turn-around time

- Depending on the number of samples, please arrange with BIOGRIP staff when to send samples (to ensure instrument availability) and preferably submit samples Monday – Wednesday for sufficient analysis time.
- Turn-around time for analysis ~ 1 week from submission date

Caution and consideration for sample submission – 'unsafe or problematic' samples

1. Samples containing aggressive chemicals such as acids or alkaline solutions can't be analysed on this instrument

2. Care should be taken when samples contain organic solvents (ethanol, methanol, DCM, acetonitrile, hexane), in some instances it may not be possible to analyse these samples using this technique – please indicate if samples contain any of these chemicals
3. Samples may not contain any explosive substances
4. During TOC/NPOC analysis the following problems may arise
    a. Samples may contain organic compounds which precipitate when the samples are pre-acidified to pH<2. For these samples the TIC/TOC/TC method will be used to prevent precipitation. Please note that this will have cost implications as this method is more expensive than the NPOC method.
    b. Samples which contain foaming agents, will also be analysed using the TIC/TOC/TC method. The NPOC method involves pre-acidifying and sparging the sample on the autosampler with synthetic air, which result in excessive foaming and spills. This may damage the instrument. Please note that this will have cost implications as this method is more expensive than the NPOC method.

Submission of 'blanks' for specific experiments

1. In the event that experiments involve **preparing extracts** please include the following sample blanks to verify purity of water and products used
    a. Ultrapure water used to prepare extraction solutions (to verify TOC, TIC, TC content of water)
    b. Extraction solution containing chemicals (to verify purity of chemicals used)

2. Method validation (optional)
    a. If experiments for example involve evaluation of breakdown or removal of specific compounds of interest, where possible please discuss with BIOGRIP staff whether standard solutions can be provided to verify combustion of products as an additional QC.

Procedures for preparing <u>solid</u> samples

1. The amount of solid material collected will depend on the sample matrix. Please collect enough volume (g) to have a representative sample.
2. Samples must be milled (using eg a ball mill or mortar and pestle) to a fine powder to a homogeneous consistency.
3. A minimum of 1 gram can be provided for analysis.
4. Label samples according to your sample submission template (downloadable on the website).

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Terminology and definitions

Total carbon – sum of the total organic and total inorganic carbon.

Total Inorganic carbon (TIC) — eg carbonate, bicarbonate, and dissolved CO_2, can be particulate or dissolved compounds.

Total organic carbon (TOC) — all carbon atoms covalently bonded in organic molecules and is the sum of the purgeable and non-purgeable organic carbon. During acidification to pH< 2 using HCl, the total inorganic carbon fraction is removed by sparging the samples with synthetic air.

Purgeable organic carbon (also called *volatile organic carbon*) — is the fraction of TOC removed from an aqueous solution via gas stripping under specified conditions.

Non purgeable (NPOC) – NPOC is regarded as equivalent to the TOC content IF the purgeable organic carbon (POC) is assumed to be negligible.

NPOC - Dissolved organic carbon (DOC) — the fraction of TOC that passes through a 0.45-µm-pore-diameter filter.

NPOC - Suspended organic carbon (also called *particulate organic carbon*) — the fraction of TOC retained by a 0.45-µm filter.