

Application Note

Simple TOC determination of sediments and soils using the suspension method

The storage of wastes containing more than 1 mass% of organic compounds (TOC) (landfill class I) or 3 mass% (landfill class II) is, according to the 'Ordinance on Environmentally Compatible Storage of Waste from Human Settlements and on Biological Waste-Treatment Facilities' (Abfallablagerungsverordnung - AbfAbIV) [1] not allowed without thermal or mechanical-biological treatment. This also applies to wastes such as soils, sediments or construction waste. Using the new suspension method, these solid materials can be analysed with clearly reduced expenditures in time and costs.

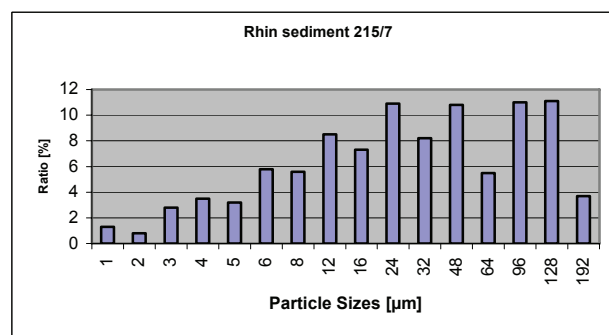


Sampling: Sediments from a harbour basin

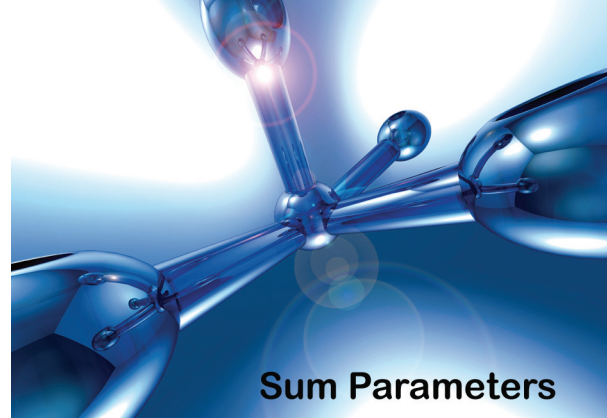
Compared to solid sample TOC, the alternate suspension method offers considerable advantages:

1. Less expenditure in time through simple sample preparation
2. Reduced probability of errors due to contamination
3. Consistent automation via the ASI-V autosampler
4. Reduced system complexity and consequently clearly reduced costs.

The preparation of the suspensions from sediments and soils requires optimisation of the sample preparation procedure. The sample material is dried and finely pulverised using an appropriate grinding method (for example ball mill). A typical particle size distribution is shown in the following diagram:



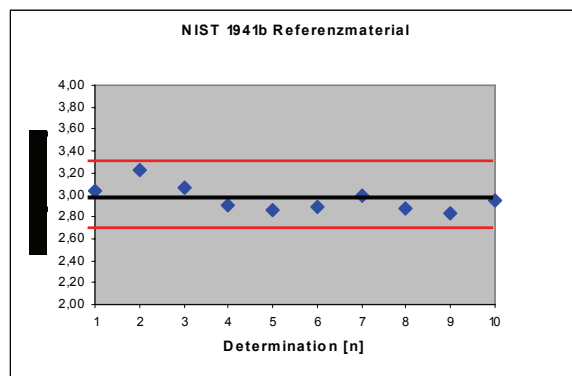
The pulverised sample is added to a 0.22 N hydrochloric acid solution (p.a. diluted with ultrapure water) in a ratio of 1:1000 and subsequently treated in a precision dispersion tool (Ultraturrax®) to obtain a suspension. The organic compounds present in the suspension are subsequently determined via the NPOC method (Non Purgeable Organic Carbon).



Volatile organic compounds can be neglected (after drying at 105 °C). The assumption NPOC = TOC is therefore valid for this application! Ideally, a Shimadzu TOC-V_{CPX} system including an ASI-V with integrated stirring unit and external sparging unit is used for NPOC measurements in suspensions.

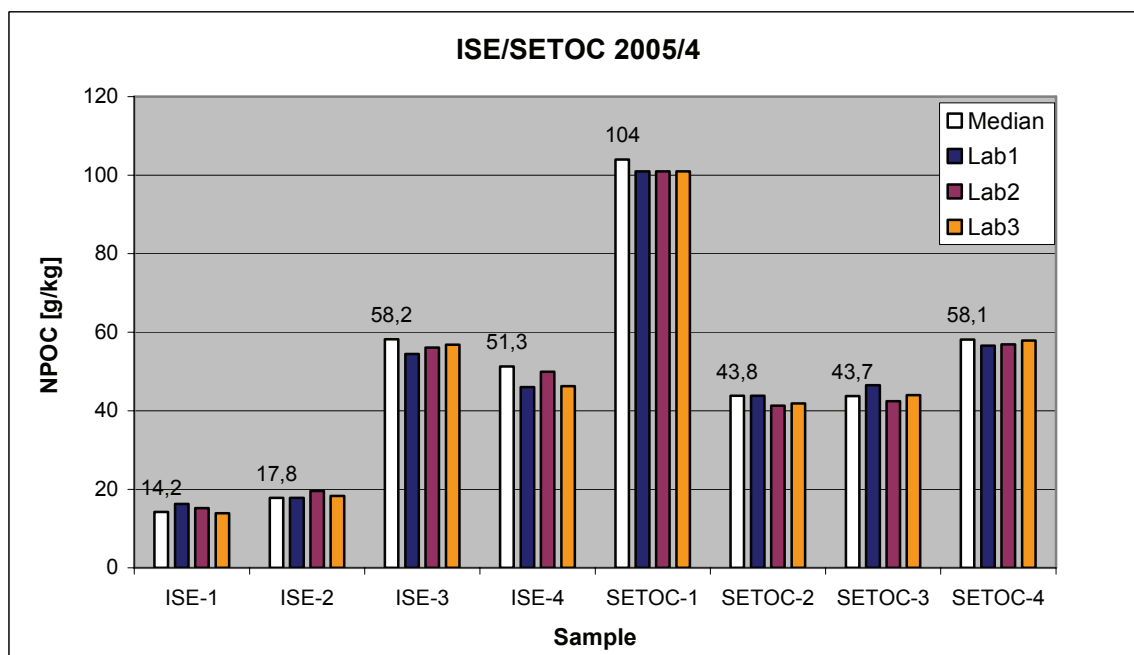
The conditions with respect to sample transport and injection correspond with the guidelines for wastewater, respectively particle tests. Combustion takes place at 720°C using a standard catalyser covered with 0.5 - 1 cm ceramic fibre. After calibration (TC) with potassium hydrogen phthalate according to DIN EN 1484, the NPOC can be quantified. At the above-mentioned dilution (1:1000) the following applies: 10 mg C/L = 1 % solid material. Smaller NPOC percentages require that a blank value for the acid be taken into account.

Using the appropriate reference material, the new suspension method can be optimally compared with the solid sample method. The following diagram shows the results of a test sequence with the 'NIST 1941b reference sediment' over a period of four months.



Round robin test:

ISE 2005/4 and SETOC 2005/4 (WEPAL, Wageningen NL)



The median is based on the results of ring tests and the number of relevant participants (ISE: 24-29; SETOC: 12-15). The participants in 'Lab 1,2 and 3' have carried out suspension method measurements independently of each other.