

METHOD STATEMENT



Determinand:

Determination of Total Organic Carbon and Dissolved Inorganic Carbon

Matrix:

Sample Types: Raw, Potable, Surface and Ground waters.

Principle of Method:

This method uses the SHIMADZU TOC-L CPH TOC analyser.

Total Organic Carbon (TOC) is the amount of carbon bound in an organic compound and it is used as a non-specific indicator of water quality. The TOC in water sources can either be derived from natural sources (eg:- Humic acid, Fulvic acid) or synthetic sources (eg:- Detergents, Pesticides).

This method can be used to determine the TOC or DOC of a sample. For a DOC measurement the sample should be filtered through a 0.45um filter disc prior to acidification with the orthophosphoric acid preservative. In this method TOC and DOC are determined by measuring the amount of Non-Purgeable Organic Carbon (NPOC). NPOC is commonly referred to as TOC.

First the sample is acidified to pH 2 - 3 and then sparge gas is bubbled through the sample to eliminate the IC component. The remaining TC is measured to determine total organic carbon; the result is referred to as TOC. Purgeable organic substances in the sample are lost during the sparging process. The instrument uses a 680°C combustion catalytic oxidation method. The carbon dioxide generated is swept by an inert carrier gas to an Infra-red detector where the concentration of the carbon dioxide present is determined. The amount of carbon present in the sample is directly proportional to the absorbance of CO₂ measured.

Sampling and Sample Preparation:

Samples are normally collected in 500 ml PET bottles. Other size PET bottles are also suitable.

If analysis cannot be immediately undertaken, samples should be stored at a temperature of $3 \pm 2^{\circ}\text{C}$ until the day of analysis. Samples should be warmed up to room temperature prior to analysis and analysed within 8 days of the sampling date.

Interferences

None

Performance of Method:

Range of Application:

LOQ - 10 mg/l C

The analytical range may be extended by sample dilution.

Limit of Quantification:

Method standardised LOQ for both TOC_P and TOC_P2 is <0.7 mg/l C

Recoveries of Compounds, Bias and Uncertainty of measurement:

TOC_P

| Sample type | Mean sample result (mg/l) | Mean sample spike result (mg/l) | Spike Recovery (%) | Bias (%) | Uncertainty (%) |
|--------------|---------------------------|---------------------------------|--------------------|----------|-----------------|
| Soft | 1.494 | 4.553 | 101.95 | - | - |
| Medium | 1.388 | 4.884 | 99.88 | - | - |
| Hard | 1.583 | 4.566 | 99.45 | - | - |
| Raw | 2.554 | 4.503 | 97.48 | - | - |
| Filtered Raw | 2.346 | 4.704 | 94.29 | | |

METHOD STATEMENT



| Sample type | Mean sample result (mg/l) | Mean sample spike result (mg/l) | Spike Recovery (%) | Bias (%) | Uncertainty (%) |
|--------------|---------------------------|---------------------------------|--------------------|----------|-----------------|
| Borehole | 0.822 | 4.841 | 100.47 | - | - |
| 2.0 mg/l Std | 2.115 | - | - | 5.73 | ±18.59 |
| 8.0 mg/l Std | 8.139 | - | - | 1.73 | ±5.53 |

TOC_P2

| Sample type | Mean sample result (mg/l) | Mean sample spike result (mg/l) | Spike Recovery (%) | Bias (%) | Uncertainty (%) |
|--------------|---------------------------|---------------------------------|--------------------|----------|-----------------|
| Raw | 1.657 | 2.822 | 101.24 | - | - |
| Filtered Raw | 1.424 | 2.933 | 100.59 | - | - |
| 2.0 mg/l Std | 2.199 | - | - | 2.96 | ±7.57 |
| 8.0 mg/l Std | 8.237 | - | - | -2.20 | ±10.86 |

References:

The Instrumental Determination of Total Organic Carbon, Total Oxygen Demand and Related Determinands 1979.

Methods for the Examination of Waters and Associated Materials. (HMSO), ISBN 011-751458 6

SHIMADZU Total Organic Carbon analyser, TOC-L CPH/CPN, User's Manual.

Water Quality-Sampling-Part 3: Guidance on the Preservation and Handling of Water Samples. BS EN ISO 5667-3-2003, Page 14.