INFORMATION

for Participants in the Interlaboratory Trial to Validate ISO/CD 21027

Water quality — Determination of total organic carbon for suspended solid containing water samples based on combined ultrasonic and alkaline extraction pretreatment

This information provides technical instructions and analytical procedures for laboratories participating in the interlaboratory trial to validate ISO/CD 21027(2nd). The procedures are based on the draft standard and the ILT planning documents (ISO/TC 147/SC 2/WG 70/N124 and N126).

1. Sample Distribution

This ILT involves the use of the following six sample types :

Sample Name	Source	Purpose	Distribution Kit and Content
ERM-CC144	Sewage sludge [a]. (TOC, 36 g/100g)	Quality control for sample pretreatment	Dried particles, individually packed, **200 mg/kit × 6 ea**
RM-A	Channel sediment[a]	ILT comparison sample	Dried particles, individually packed, **100 mg/kit × 6 ea**
RM-B	Algae[a]		
$Wastewater_1$	Sewage influent		
$Wastewater_2$	Wastewater A[b]		
$Wastewater_3$	Wastewater B[b]		

a) RM(reference material) samples are provided without additional pretreatment after procurement.

b) Wastewater particulate samples were prepared via solid-liquid separation, drying, and sieving through a 1.00 mm mesh. Homogeneity and short-term stability were verified per ISO 13528 and ISO 33405.

2. Reagents and Equipment

(Refer to ISO CD21027 Clause 6 and Clause 7)

Water *(6.1)*

NaOH (1 M): for alkaline extraction *(6.2)*

HCl (1 M): for pH neutralization *(6.3)*

Potassium hydrogen phthalate (KHP): TOC and TC standard *(6.8.2)*

Sodium carbonate/bicarbonate: TIC standard *(6.8.6)*

Cellulose $(C_6H_{10}O_5)_n$: for suspended solid control (*6.8.8)*

Sonicator*:* for pretreatment (Bath or Probe type) (*7.1)*

TOC Analyzer: HTC or COHR-based detection approaches *(7.3)*

Magnetic Stirrer: for sample mixing *(7.3.3)*

3. Key Analytical Procedures (ISO CD21027)

This procedure outlines the core steps for TOC analysis using the difference method (TC - TIC) following CUAL pretreatment based on ISO/CD 21027(2^{nd}) [*document* N124], including preparation of suspended solid samples (SS, 200 mg/L) from various environmental origins (e.g., wastewater, algae, sediment, etc.).

***Note** on Sample Injection in ISO/CD 21027*(2^{nd})*: The term replicate injections, as stated in Clauses 8.2, 8.3, and 11.3, refers to "**independent replicate measurements**", meaning a single injection per independently prepared batch, rather than multiple injections from the same vial.*

For further clarification, refer to this manual and the ILT planning presentation material (document N126).

3.1 Calibration Curve Preparation

(Refer to ISO CD21027 Clause 6.8 and Clause11.2)1

Prepare standard solutions using KHP and TIC standards.
- TC standard: 1–200 mg-C/l *(6.8.10.1)*
- TIC standard: 1–200 mg-C/l *(6.8.10.2)*

Measure standards at various concentrations and construct a calibration curve within the range of TOC 0-200 mg-C/l *(11.2)*
Note 1*: The standard solutions may be used as a TC/TIC mixture, depending on the TOC analyzer manual.*
Note 2*: Use the average of at least two replicate injections for the reported TOC value.*

3.2 System check

(Refer to ISO CD21027 Clause 8.1)

Measure standard solutions covering approximately 20 % to 80 % of the calibration range.
Required criteria: Recovery > 90%, the repeatability variation coefficient (CV) < 10%
Note*: Use the average of at least two replicate injections for the reported TOC value.*

3.3 Particle processing control for TOC with cellulose particle

(Refer to ISO CD21027 Clause 8.2 and Clause 11.4.2)

Prepare cellulose suspension: 100 mg-C/l *(6.8.8.1)*
- The suspension is stirred using a magnetic stirrer until it becomes homogeneous.
- Ultrasonication should not be used because it reduces the particle size.

Test for particle processing control
- During analysis with an autosampler, continuous stirring is required.
- The mean value shall be calculated from **three independent replicate measurements**, each based on a single injection per batch.

Quality Control criteria: Recovery > 90%, the repeatability variation coefficient (CV) < 10%

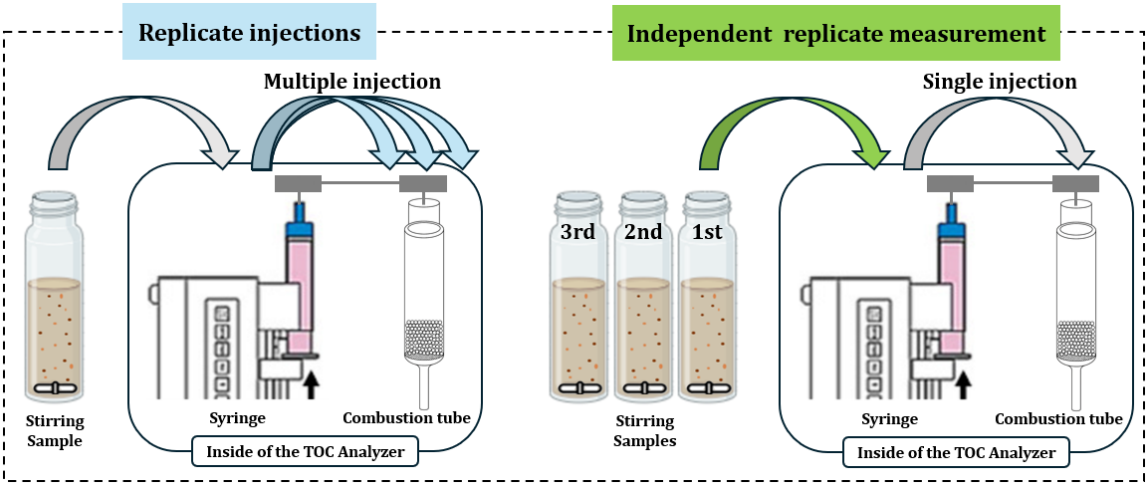

Figure 1 Comparison of Sample Injection Methods: Single vs. Multiple Injection

***Note**: Independent replicate measurements (i.e., single injections) are performed using only the first injection from each batch sample. This approach is intended to minimize the influence of particle settling inside the syringe during oxidation of the previous sample. For example, to obtain three replicate measurements, three separate batches should be prepared, and only the first injection from each batch should be used.*

3.4 Sample pretreatment control for TOC with particles of environmental origin

(Refer to ISO CD21027 Clause 8.3, Clause 10.1, and Clause 11.4.3)

Note : Please refer to the guide video via the following link along with this manual: https://youtu.be/LSENgPZVcDg

① Suspension Preparation

- Dissolve 40.0 mg of reference particle (ERM-CC144) into 200 mL of water to create a 200 mg/L suspension.
- Prepare three independent suspensions for triplicate measurements.
- Stir the suspension for 10 minutes before pretreatment.

② CUAL Pretreatment

- Add 10 mL of 1 M NaOH to the stirred suspension (1/20 volume ratio).

***Note**: Most samples generally reach a pH range of 11 to 13 after the alkaline extraction step.*

- Immediately perform ultrasonic treatment:

***Note 1**: Sonication times are typically in the range of 30 to 40 minutes for bath-type and 5 to 10 minutes for probe-type devices (adjust to achieve TOC recovery of at least 70% for the QC samples (ERM-CC144).*
***Note 2**: Bath is good for multiple samples; Probe is efficient for single samples.*
***Note 3**: The sample container shall be sealed during Bath-type sonication to prevent contamination.*

③ pH Neutralization

- After sonication, add 10 mL of 1 M HCl(1/20 volume ratio), as with 1 M NaOH injection.

***Note**: This process is intended to facilitate the adjustment of pH to below 2 during the subsequent acid addition step for inorganic carbon measurement.*

④ Sample Injection

- During analysis with an autosampler, continuous stirring is required.
- The final TOC value shall be calculated as the mean of three independent replicate measurements, each based on a single injection from a separately prepared batch.

⑤ TOC Measurement and Result Calculation

- Analyze using the TC-TIC method
- Result Calculation:

$$TOC_{result} = (TOC_{raw} - TOC_{Blank}) \times DF$$

***Note 1**: The TOC value of the water sample used in the preparation of the suspension shall be referred to as TOC_{Blank}*
***Note 2**: The dilution factor (DF), resulting from the addition of NaOH and HCl, shall be applied in the calculation of TOC_{result}.*

⑥ Result Validation

Required Criteria: Recovery > 70%, the repeatability variation coefficient (CV) < 20%, both calculated from the mean of three independent replicate measurements, each derived from a single injection per batch.

***Note**: While the CV is generally expected to be less than 20%, the final acceptance criterion will be determined based on the results of this interlaboratory trial.*

3.5 CUAL pretreatment and TOC analysis for environmental samples

(Refer to ISO CD21027 Clause 10.1 and Clause 11.4)

Note : : Please refer to the guide video via the following link along with this manual: https://youtu.be/LSENgPZVcDg

- Apply the same procedure (Steps ① to ⑤) to RM-A, RM-B, and Wastewater 1–3 samples.
- Each sample shall be analyzed in triplicate (n = 3), using independently prepared batches.

4. Reporting of Results

Record results in the attached Excel file (Appendix. ILT Data sheet.xlsx).

Include details on instruments and analytical procedures as requested.

Submit the completed file via email to the ILT organizer (hyuns@seoultech.ac.kr).

Note: *The results of the interlaboratory trial will be provided to all participants in an anonymized format. Following ISO 5725-2, each participating laboratory will receive a unique laboratory code, which can be used to identify its results in the final report. Comprehensive summary tables and graphical evaluations will be sent via email in the form of attached PDF files.*