

USGS Troy WSC Laboratory 425 Jordan Road Troy, NY 12180	Total Carbon and Total Nitrogen SOP	
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USGS New York Water Science Center  
Soil and Low Ionic Strength Water Quality Laboratory  
**Total Carbon and Total Nitrogen**  
Standard Operating Procedure

## 1. Scope and Application

### 1.1 Analytes

Total Carbon and Total Nitrogen

### 1.2 Reporting Limit

None

### 1.3 Applicable Matrices

This method is used to determine total carbon and total nitrogen in soils.

### 1.4 Dynamic Range

0 % to 100 %

## 2. Summary of Procedure

Prior to analysis, sieved soil samples are weighed into tin capsules using a microbalance. An autosampler introduces the samples to a high temperature reactor, where they are completely combusted in an O<sub>2</sub> environment. Following combustion, the gases produced, CO<sub>2</sub> and N<sub>2</sub>, are carried by helium flow to columns where they are separated chromatographically, and finally eluted to a series of three thermal conductivity detectors for quantification.

## 3. Safety Issues

### 3.1 Chemical Hazards

- A. Gloves, safety glasses, and lab coats should be worn when preparing and performing this analysis.
- B. For proper handling techniques for specific chemicals, consult the appropriate Material Safety Data Sheets (MSDS).

### 3.2 High Temperature Hazards

- A. Due to the high temperatures produced by the furnaces in this elemental analyzer, care must be taken when performing maintenance. Use heat resistant gloves, or shut the instrument down and allow to cool to room temperature.

## 4. Sample Preparation, Storage, Processing and Analysis Times

### 4.1 Sample Preparation and Storage

All soil samples are air-dried, then sieved, and stored at room temperature in plastic zip lock bags. As required for the elemental analyzer, O-horizon samples pass through a 1-mm sieve, while all other horizons pass through a 2-mm sieve.

### 4.2 Processing and Analysis Times

Lab analysis, including sample preparation: 6 months

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LIMS entry: one week

## 5. Reagents and Standards

### 5.1 General Information

All standards are commercially purchased and should be stored in the original container. Date the standard containers when received, and date when opened. Note expiration date, if any.

### 5.2 Standards

#### A. Standards

1. Atropine, purchased from CE Elantech.
2. Store tightly capped at room temperature.

#### B. QC Samples

1. Atropine, purchased from CE Elantech. The QC material must be from a different lot from the standard material.
2. Store tightly capped at room temperature.

#### C. Reference Materials

1. Soil NC Reference material, purchased from CE Elantech
2. In-house reference material, collected in large quantity, dried, well mixed, sieved, and stored at room temperature.

## 6. QC Procedure

- A. Quality control samples are analyzed at the start of the run.
- B. The calibration is acceptable when the measured QC values are within +/- 5% of the response factor determined at calibration.
- C. If one of the QC samples fails the acceptance criteria, recalibrate the instrument.

## 7. Chemical Analysis Procedure

### 7.1 Instrumentation and Materials

Hach Model 2100 AN Turbidimeter  
30 mL, matched sample cells  
Silicone oil  
Oiling cloth  
Lap wipes

### 7.2 Start-Up

- A. The turbidimeter should be left on 24 hours a day.

### 7.3 Calibration

- A. Perform quarterly or as needed, otherwise proceed to Step 7.4.
- B. Insert the USEPA filter module.
- C. Press **CAL/Zero**.

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- D. Wipe the StablCal <0.1 NTU standard clean and apply a thin film of silicone oil and wipe sample cell with oiling cloth. Wipe excess oil off with lab wipes. Place the cell in the cell holder using the alignment mark and close the cell cover.
- E. Press **Enter**. The instrument display counts down from 60 to 0 and then makes a measurement. This result is stored and used to determine a correction factor.
- F. The instrument displays the expected NTU value, for the next standard and the standard number is 01 shown in the mode display. Remove the cell from the instrument.
- G. Use the well mixed 20 NTU StablCal standard next. Do not shake the sample cell. Mix by gentle inversion. Apply a thin film of silicone oil and wipe sample cell with oiling cloth. Wipe excess oil off with lab wipes. Place the cell in the cell holder using the alignment mark and close the cell cover.
- H. Press **Enter**. The instrument display counts down from 60 to 0 and then makes a measurement. The instrument applies a correction factor to compensate for the turbidity of the zero standard.
- I. Repeat steps F-H for each standard 02-05 in ascending order. The instrument increments back to 00 automatically and the previously measured zero standard value is displayed.
- J. Press **Cal**. The instrument stores the new calibration and returns to the measurement mode.

#### 7.4 Analysis

- A. Take samples out of the refrigerator and allow to equilibrate to room temperature.
- B. Verify that automatic ranging is enabled by pressing the **Range** key.
- C. Verify that **Signal Averaging** (default of 10 seconds) is enabled.
- D. Verify that **Ratio mode** is enabled.
- E. Verify that NTU unit measurement mode is enabled by pressing the **Units/Exit** key.
- F. Verify and record instrument calibration using the Gelex Secondary Standards. If the readings of the secondary standards are not within +/-5 percent of the values assigned at calibration, recalibrate the instrument with the StablCal standards.
- G. In the **Sample Mode**, use arrow key to select sample 1.
- H. Invert sample several times, pour small amount into sample cell to rinse.
- I. Invert sample again and fill sample cell to line (approx. 30 mL).
- J. Handle the sample cell by the top. Cap the sample.
- K. Hold the sample cell by the cap and wipe off, to remove water spots and finger prints.
- L. Wipe all moisture from the outside of sample cell and then wipe sample cell with oiling cloth. Wipe excess oil off with lab wipes.

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- M. Invert sample in sample cell and place the cell in the cell holder using the alignment mark and close the cell cover.
- N. Press **Enter** which clears the stored data buffer.
- O. The instrument will measure the turbidity of the sample in the cell holder and a value will be displayed. Press **Print**. The measurement will be printed using the internal instrument printer.
- P. Using the arrow keys, toggle to sample 2.
- Q. Rinse previously used sample cell and cap three times with Milli-Q water and repeat steps 7.4.H to 7.4.P for each of the remaining samples.
- R. When samples are completed, rinse sample cell three times with Milli-Q water and store filled with Milli-Q water.

## 7.5 Shut Down

- A. Close the cell holder cover and cover the instrument with the dust cover.

## 7.6 Maintenance

- A. Before each calibration, gently blow out sample cell holder with compressed air.
- B. Before each calibration, clean the sample cell(s) that are used for analysis in the ultrasonic cleaner.

## 7.7 Data Processing and LIMS Entry

- A. Create an Excel spreadsheet using the data from the turbidimeter printer. In column A, type in the SSN and in column B, type in the respective turbidity values.
- B. Save as a comma delimited file (.csv file extension) under \\Igsahaewgs044\lab\_data\turbidimeter in the Edited Data file folder using the mmddyyyy format.
- C. Print a copy, note the filename on the copy, and close the file.
- D. Proofread the file to insure there aren't any data entry mistakes.
- E. Double click **Watershed LIMS** icon.
- F. Click **Custom**, then **Import Data**.
- G. Under the Import drop down, choose **pH or Cond or ANC**.
- H. Choose and open the **turbidimeter** folder and the file to be imported.
- I. Choose **Client, Analysis, Units**, type in **Test Date**, and choose **Analyst**.
- J. Exclude and/or edit any data necessary.
- K. Click **Client ID to Sample No**.
- L. Click **Set Data**.
- M. Investigate problems for data that did not transfer or are duplicated.

## 8. Archiving

### 8.1 Data

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Data files are backed-up daily by an automatic back-up program. Hard copies of the runs are filed and kept indefinitely. The laboratory LIMS system is backed up daily by an automatic back-up program.

## **8.2 Samples**

Samples are returned to fridge for further processing. Sample bottles are cleaned and reused for new samples.

## **9. References**

Hach Model 2100 AN Laboratory Turbidimeter, 2000, Instruction Manual, Hach Company.

## **10. Key Words**

Turbidity, water