Water Quality
Procedures and Practices Manual

PREPARED FOR:

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INGHAM COUNTY DRAIN COMMISSIONER

PREPARED BY:

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WATER QUALITY PROCEDURES AND PRACTICES MANUAL
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1. FOREWORD

The National Pollutant Discharge Elimination System (NPDES) Program protects the surface waters of the state by assuring that discharges of wastewater comply with state and federal regulations. Anyone discharging or proposing to discharge wastewater to the surface waters of the state shall make application for and obtain a valid NPDES MS4 permit prior to the wastewater discharge. As part of the NPDES Program, the Illicit Discharge Elimination Plan (IDEP) is utilized to ensure that any of points of discharge into the surface waters of the state are in compliance. Outfalls are inspected by field personnel for presence of flow, depth, surrounding area land usage, odor, color, turbidity, stains, vegetation, and a variety of chemical water quality tests. The following document describes the initial outfall screening process and the subsequent testing procedures for ISCO continuous monitoring equipment, total solids, total suspended solids, total phosphorus, total nitrogen, nitrate-N, turbidity, and the infiltrometer.

2. WATER QUALITY SAMPLING PROCEDURE

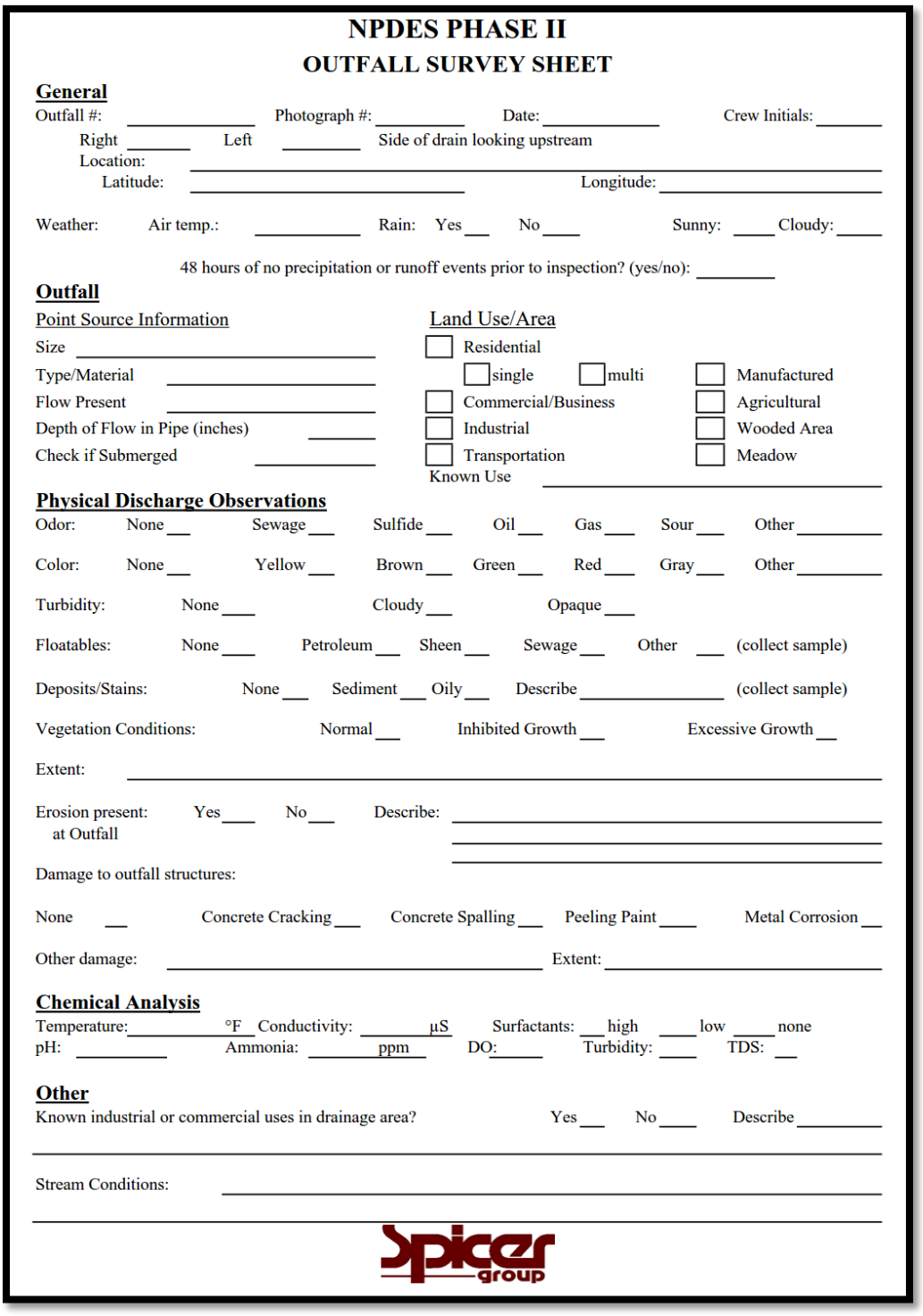
A. Water Sampling Procedure

1. Inspection

Initial screening of outfalls include making the following observations:

- Size of pipe
- Material
- Presence of Flow
- Land Use
- Odor
- Color
- Turbidity
- Floatables
- Deposits/Stains
- Vegetation Conditions
- Erosion
- Damage
- Temperature
- Conductivity
- Surfactants
- pH
- Ammonia
- Dissolved Oxygen
- Turbidity
- Total Dissolved Solids
- Stream Conditions

An example of a checklist for initial screening is seen below:



NPDES PHASE II

OUTFALL SURVEY SHEET

General

Outfall #: _____ Photograph #: _____ Date: _____ Crew Initials: _____

Right _____ Left _____ Side of drain looking upstream

Location: _____

Latitude: _____ Longitude: _____

Weather: Air temp.: _____ Rain: Yes ___ No ___ Sunny: ___ Cloudy: ___

48 hours of no precipitation or runoff events prior to inspection? (yes/no): _____

Outfall

Point Source Information

Size _____

Type/Material _____

Flow Present _____

Depth of Flow in Pipe (inches) _____

Check if Submerged _____

Land Use/Area

☐ Residential ☐ single ☐ multi

☐ Manufactured

☐ Commercial/Business ☐ Agricultural

☐ Industrial ☐ Wooded Area

☐ Transportation ☐ Meadow

Known Use _____

Physical Discharge Observations

Odor: None ___ Sewage ___ Sulfide ___ Oil ___ Gas ___ Sour ___ Other _____

Color: None ___ Yellow ___ Brown ___ Green ___ Red ___ Gray ___ Other _____

Turbidity: None ___ Cloudy ___ Opaque ___

Floatables: None ___ Petroleum ___ Sheen ___ Sewage ___ Other ___ (collect sample)

Deposits/Stains: None ___ Sediment ___ Oily ___ Describe _____ (collect sample)

Vegetation Conditions: Normal ___ Inhibited Growth ___ Excessive Growth ___

Extent: _____

Erosion present: Yes ___ No ___ Describe: _____
at Outfall

Damage to outfall structures:

None ___ Concrete Cracking ___ Concrete Spalling ___ Peeling Paint ___ Metal Corrosion ___

Other damage: _____ Extent: _____

Chemical Analysis

Temperature: _____ °F Conductivity: _____ µS Surfactants: ___ high ___ low ___ none

pH: _____ Ammonia: _____ ppm DO: _____ Turbidity: ___ TDS: ___

Other

Known industrial or commercial uses in drainage area? Yes ___ No ___ Describe _____

Stream Conditions: _____

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After initial inspection on the outfall is complete, water samples can then be collected via grab sampling methods. In order to collect a grab sample, field personnel must first wear clean nitrile gloves in order to avoid contamination of samples and to protect the personnel from potentially hazardous sample water. Being sure not to disturb any bottom sediments or surrounding vegetation, the sample bottle is then placed mid-flow in order to capture a quality water sample from the outfall. Once capped, the sample must be labeled properly according to where the sample was collected, what time it was collected, and what type of analysis is to be performed on the sample. After it is labeled, the sample will immediately be placed on ice in a cooler until it is transported back to the laboratory for analysis.

2. Chain of Custody

A chain of custody sheet should also be filled out before submittal of samples to a laboratory. A chain of custody serves as a log of all personnel involved from the collection of ample to the testing of the sample. The chain of custody procedure is as follows in the next pages.

Chain of Custody Procedures

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Chain of Custody Procedures

- Purpose is to keep a tracking log of all the people that are involved from collection of the sample to testing of the sample
- Samples should be placed in an appropriate transfer container, labeled and delivered to the analyzing laboratory.
- When samples are being transported, each person that the samples are transferred to should sign the chain of custody.

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Chain of Custody Procedures

- The field team leader will insure that the chain of custody form is properly filled out.
- After the samples are relinquished by the field team leader the chain of custody form is signed by the person at the laboratory receiving the samples.
- If there are any other transfers of the samples they must be signed by the person relinquishing the samples to the person receiving them.
- An example of a filled out Chain of Custody form is shown on the following slide.

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Example of a filled out Chain of Custody form

Chain of Custody

Client Name: INGHAM COUNTY
Contact Person: TIM INMAN
Project Name/Number TOWAR GARDEN
Date Originated: 11/21/2011

Sample Types
(SW) Surface Water
WW- Waste Water
GW-Ground Water
X- Other (_____)

							Parameters									
Sample ID	Date	Time	Sample Descriptor	Sample Type	# of Containers	Preserved (Y/N)	TP	TN	TSS	TDS	TS					Notes
1, 5	11/21/11	10:45 AM	SAMPLE NO, 1	SW	1	Y	X	X	X							RETURN SAMPLE IF POSSIBLE

Comments:

Relinquished By:	Date/Time	Received By:
[signature]	11/21/2011	[signature]

Chain of Custody #

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B. Water Testing Procedure

Once in the laboratory, samples collected from various outfalls are ready for analysis. The following water testing procedures are outlined in the succeeding pages: ISCO continuous monitoring equipment, total solids, total suspended solids, total phosphorus, total nitrogen, nitrate-N, turbidity, and infiltration.

1. ISCO

a. ISCO Maintenance and Data Collection Procedures

ISCO Maintenance and Data Collection Procedures

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Pre-Collection Variables

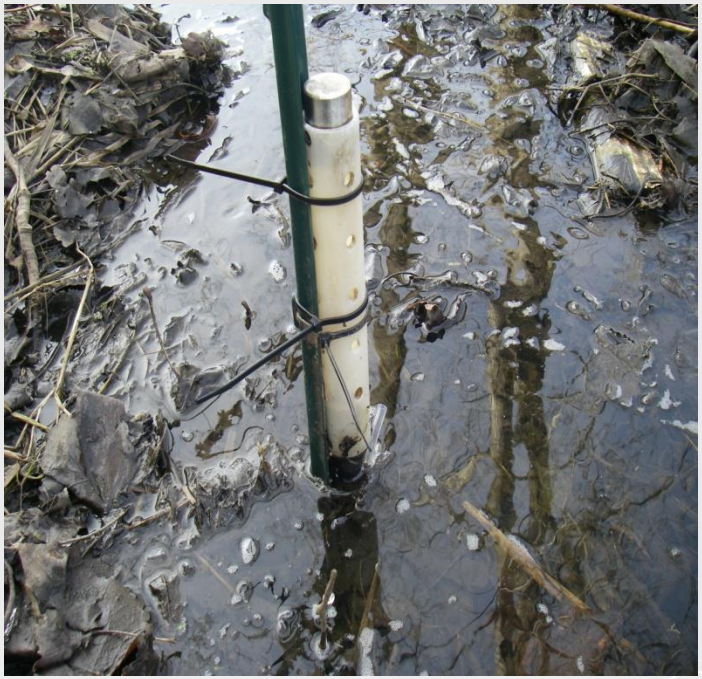
Before samples are collected the following should be recorded:

- Weather (Outside temperature, amount of rainfall, seasonal impacts)
- Select water parameters to test for
- Hold times for selected tests
- What equipment is required for tests
- Expected range of units

Site Inspection

- Inspect strainer condition and water level
- Inspect sampler condition for possible tampering
- Visual inspection of bottles

Inspection of strainer



- Inspect the strainer to make sure no debris is clogging any of the strainer openings
- If any kind of debris is trapped on the strainer remove so the ISCO pump can pull liquid to collect a sample

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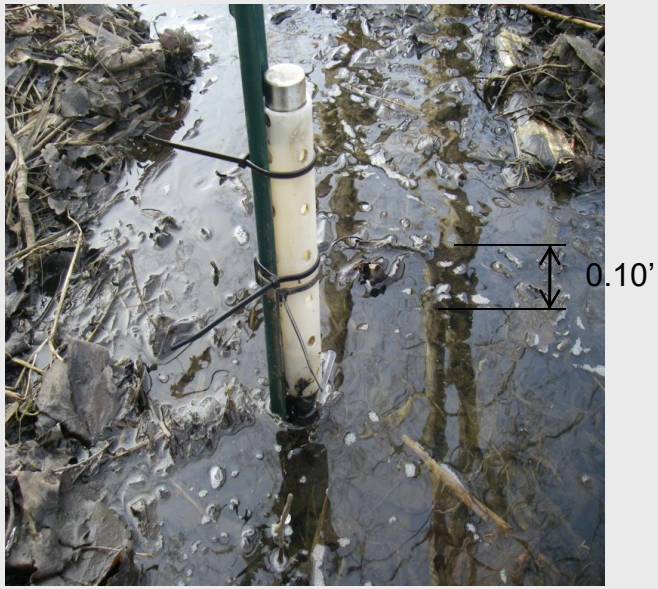
Measure the water surface elevation



- Measure the distance from the tip of the bubbler tubing to the water surface elevation.
- This will be a check later to make sure the bubbler module is operating correctly
- Under Ideal conditions the strainer and the bubbler tubing will be below the water surface elevation. In this case the water surface elevation was low.

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Visual Inspection of the ISCO Sampler

- Do a quick visual check and make sure nothing has been tampered with or is disconnected.
- Once this has been done then proceed to unlock chains to access the ISCO controller

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Procedures to collect data from the ISCO sampler

- When accessing the controller if the program is running then proceed to stop the program so the data can be extracted to a laptop.
- If the program has cycled through the 24 bottle collection program then you do not need to stop the program

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Procedures to collect data from the ISCO sampler

- Connect the data cable from the ISCO controller to the laptop to start the downloading process

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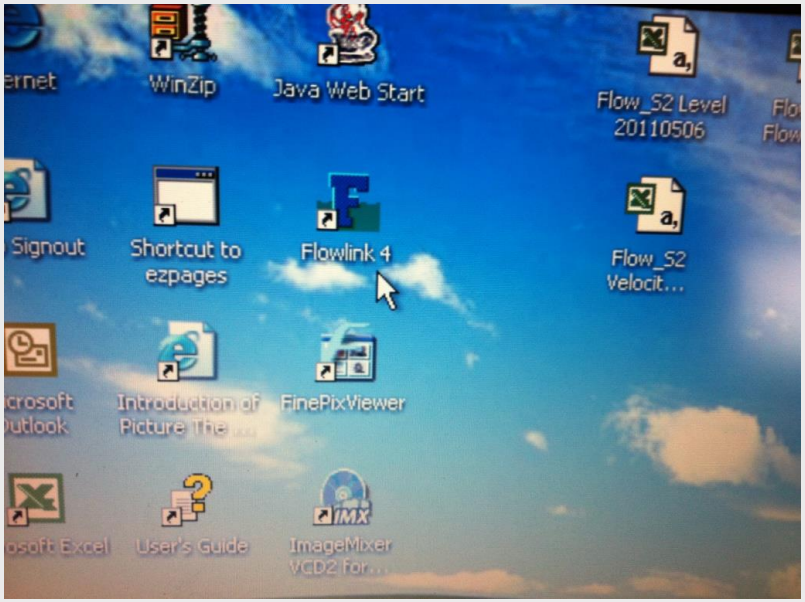
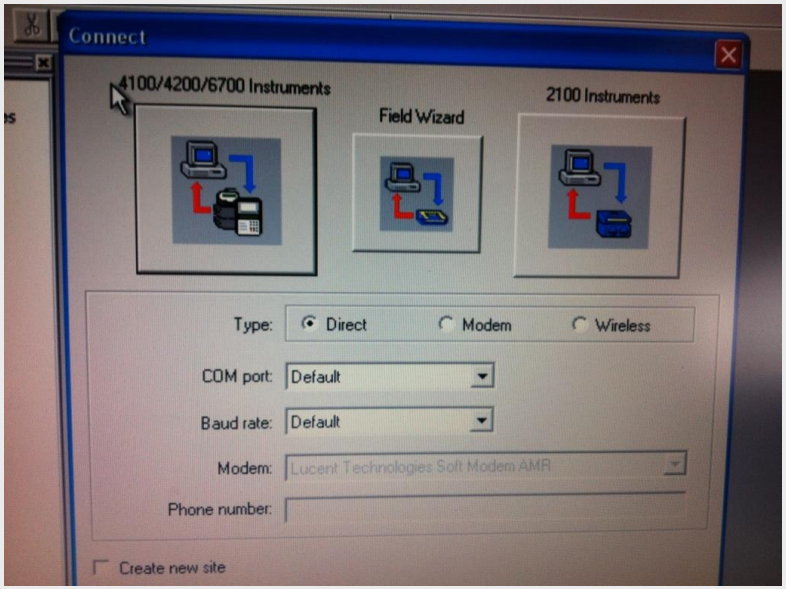
Procedures to collect data from the ISCO sampler





- Access Flowlink software from a laptop
- Once in Flowlink the quick connect screen above will appear.
- Then click on the 6700 Instrument button to start the connection process.
- Once Flowlink is connected to the ISCO Unit then in the menu tab under Actions the user will have to Retrieve Data.

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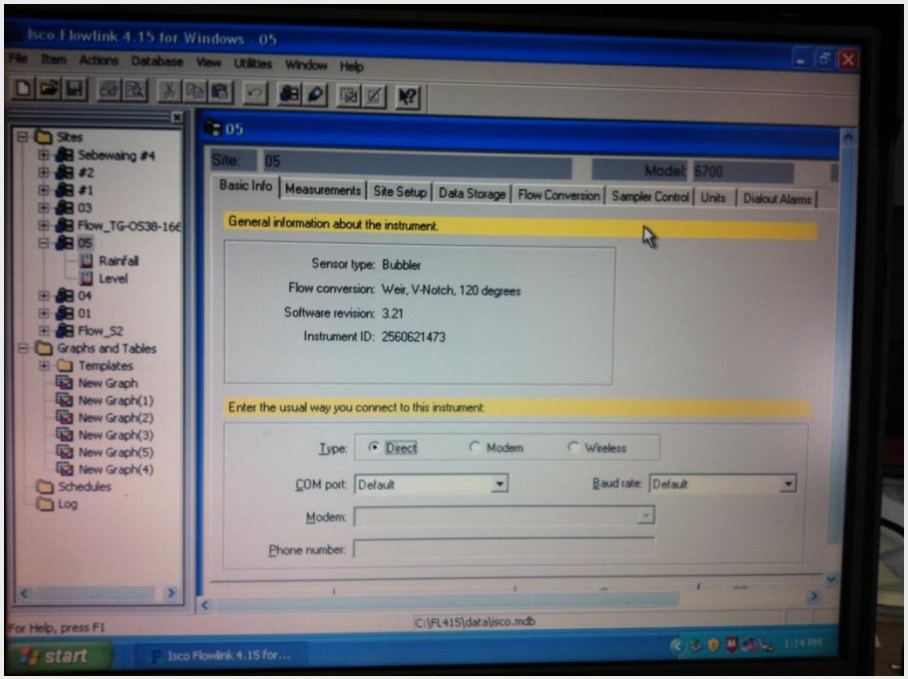
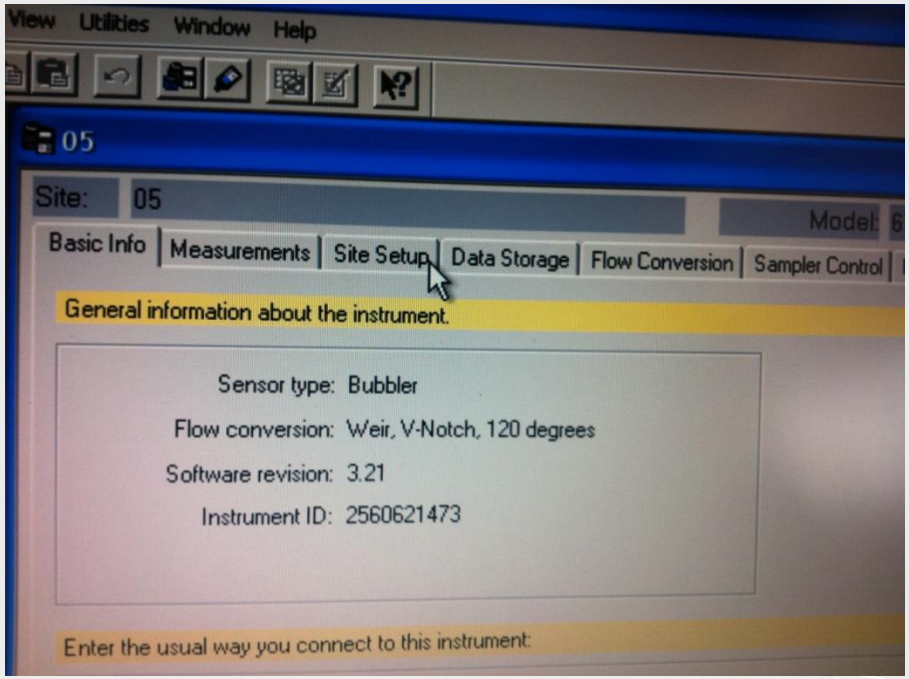
Procedures to collect data from the ISCO sampler





- After Flowlink has retrieved the data the screen to the left appears.
- To access the Site Setup Report click on the tab Site Setup as shown above right.

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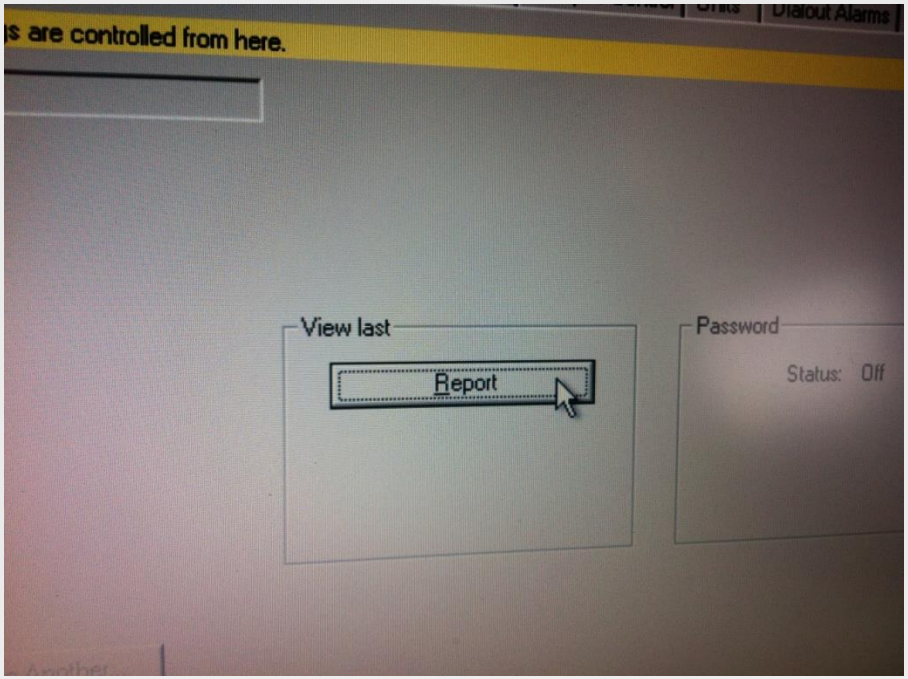
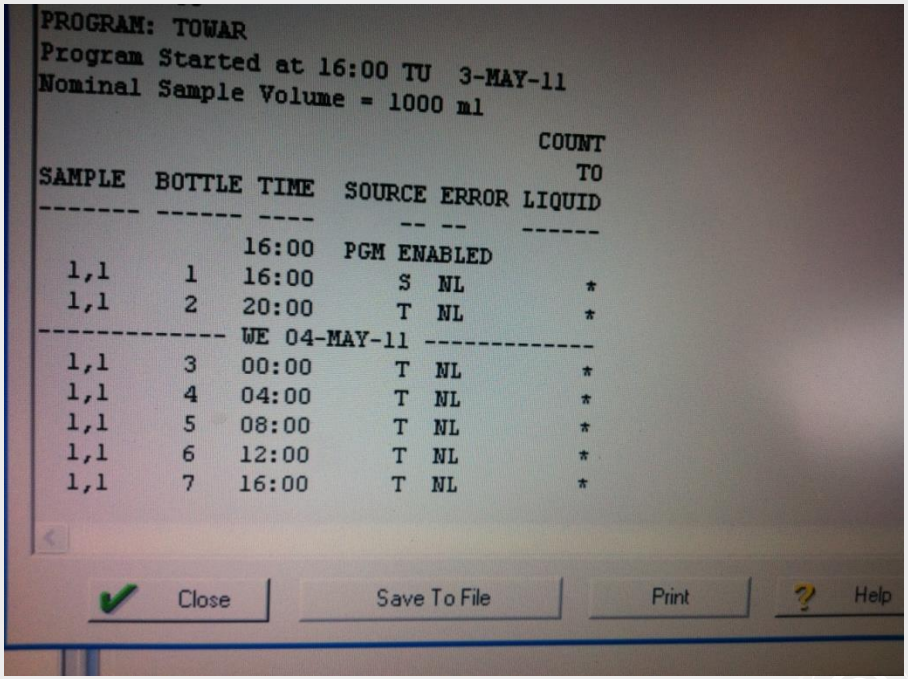
Procedures to collect data from the ISCO sampler





- After clicking the Site Setup tab it will bring up the screen to the left.
- Click the Report tab and then the Site Setup Report displays. Once displayed the user can save the file as a text file or print the report.

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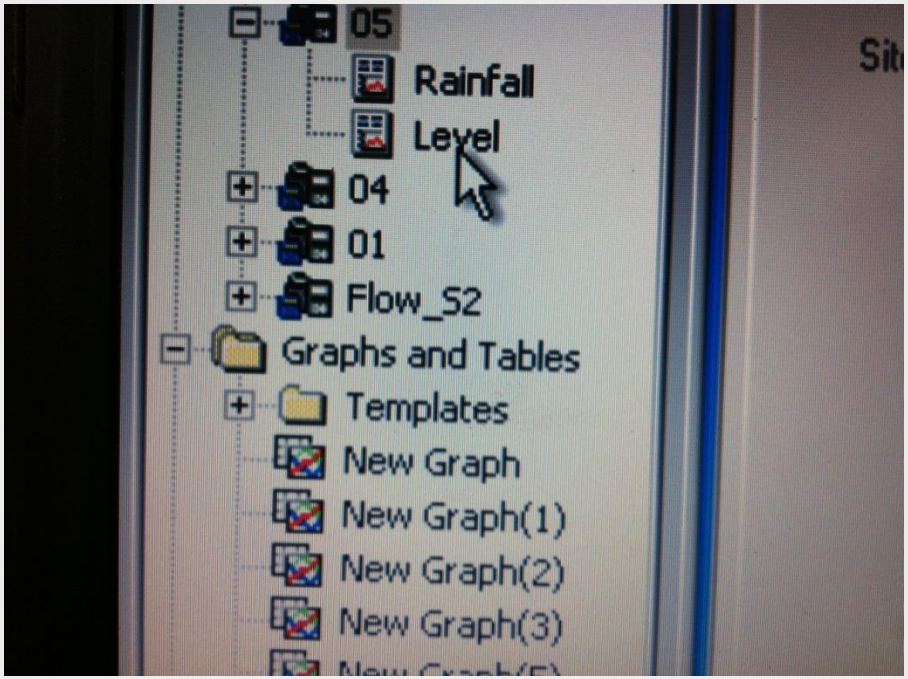
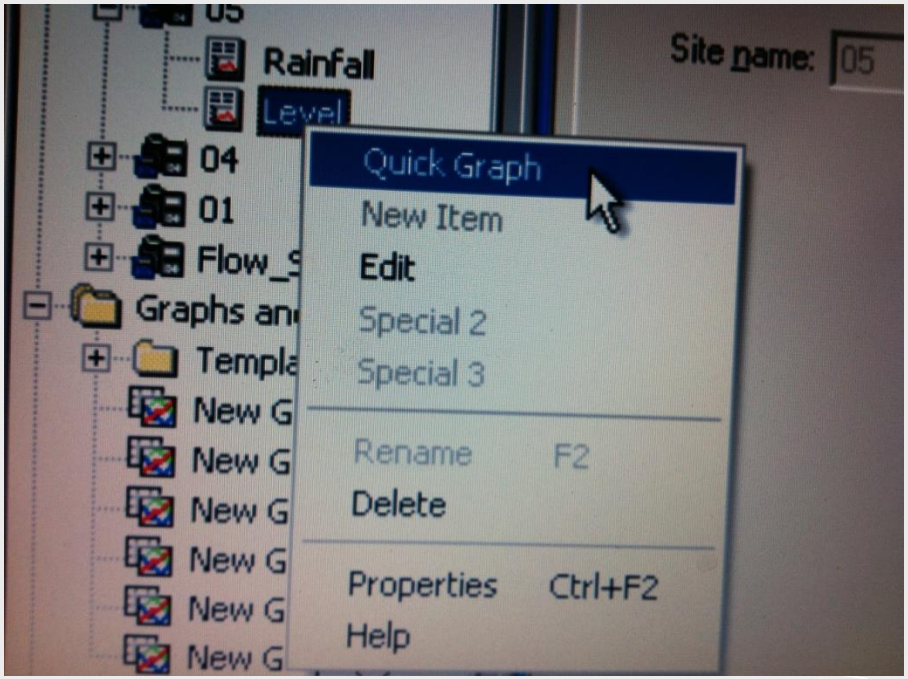
Procedures to collect data from the ISCO sampler





- To obtain data from the bubbler module expand the site by clicking the plus button to the left of the ISCO Site.
- Now right click on the level and the window to the right will appear.
- Click on the quick graph to access the bubbler data for the level

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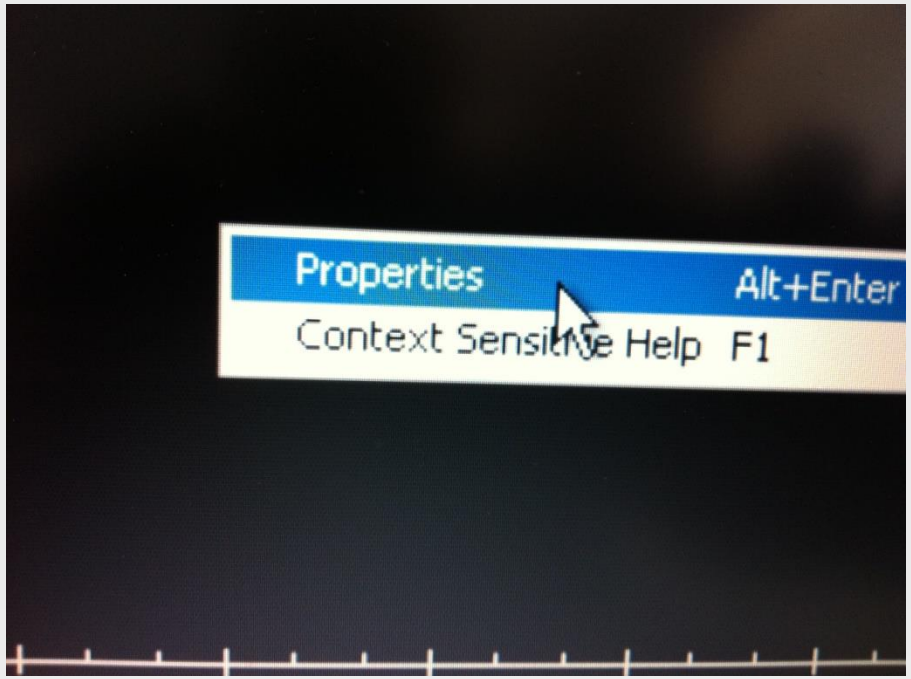
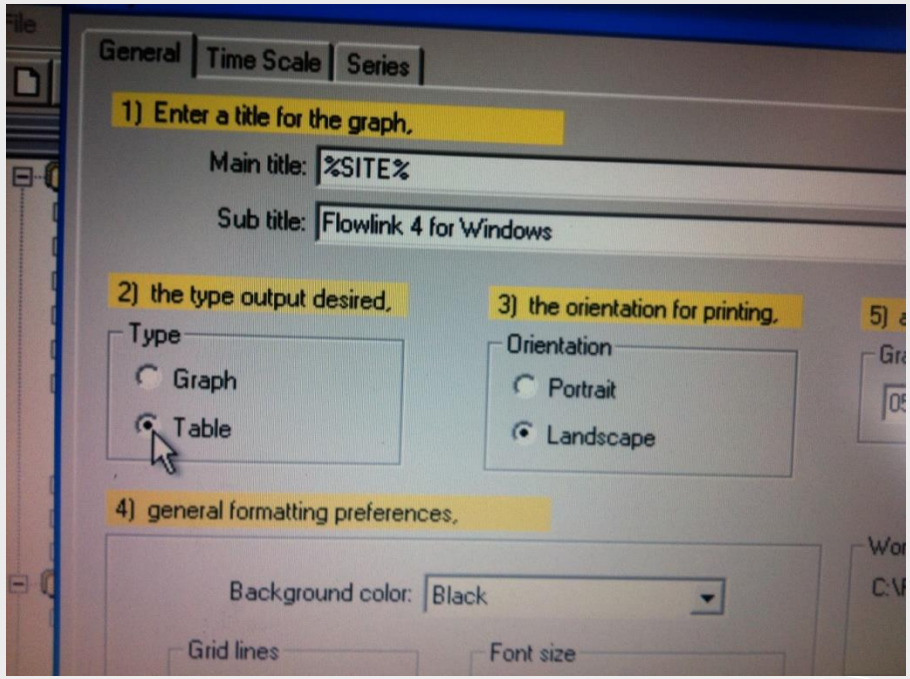
Procedures to collect data from the ISCO sampler





- Once in the quick graph view, right click any where in the graph and click Properties.
- The properties dialog box to the left appears. If the output desired is a comma separated values file then the Table button will have to be clicked.

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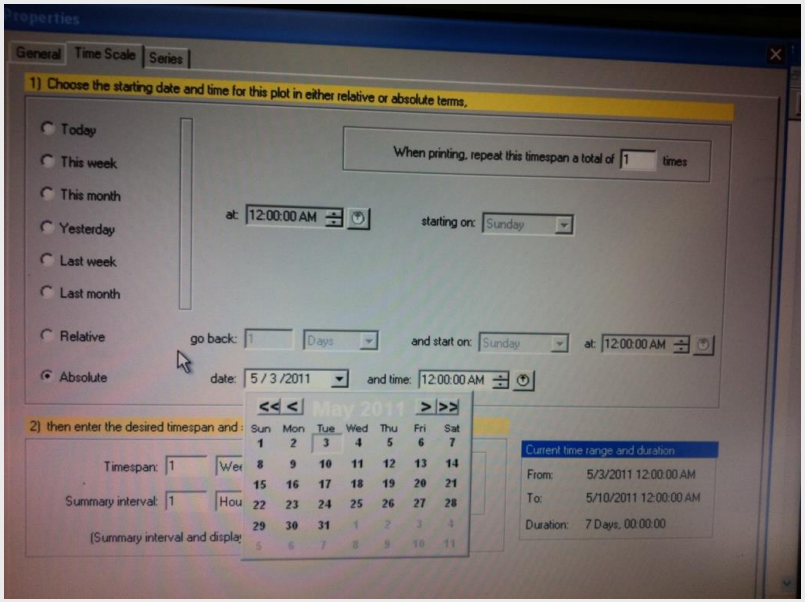
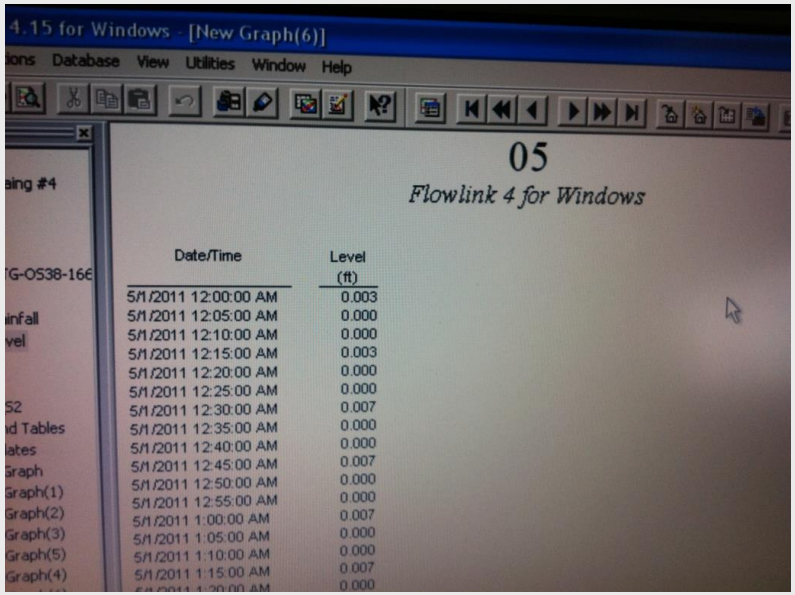
Procedures to collect data from the ISCO sampler





- Once the Table output is selected then choose the Time Scale of the desired data output.
- To select a starting date and time for the data output the Absolute option works nice. This way the user sets the exact start date and time they would like.
- Once the Table output, date and time has been entered then on some computers the users might have to drag the window up to click ok. The output form is shown above right.

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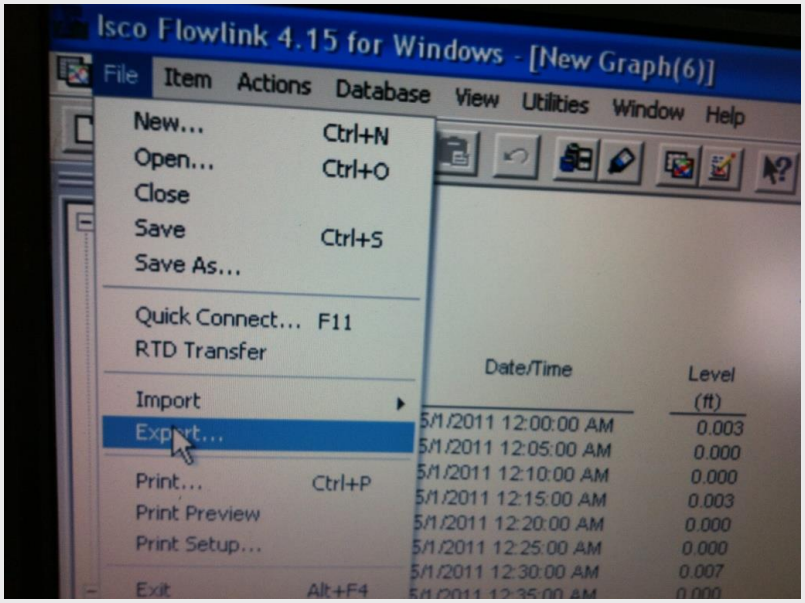
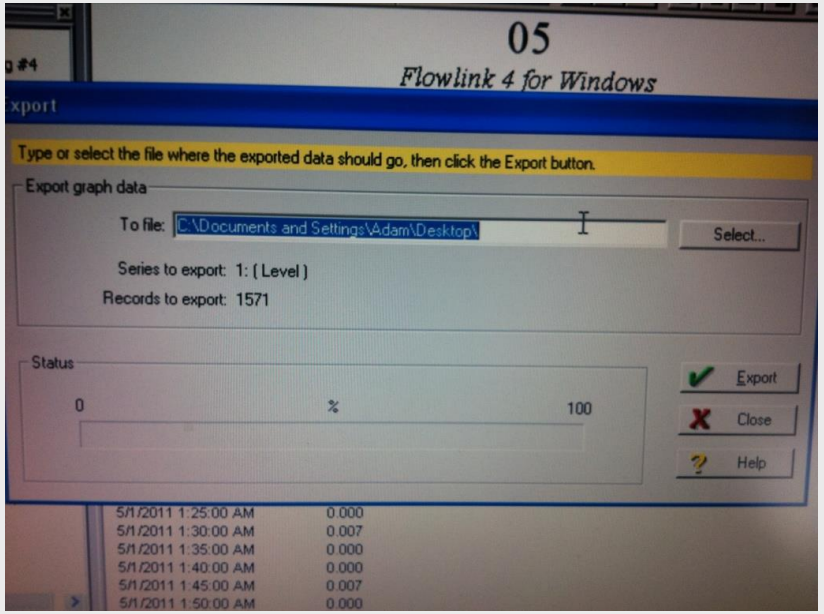
Procedures to collect data from the ISCO sampler





- After the data is displayed in the table form now the user can click on the File tab and then click Export to export the data as a comma separated value file.
- Once in the Export dialog box now the user specifies the file name and where they would like it to be saved.
- After the comma separated file is saved then the file can be converted to an Excel file and graphed.

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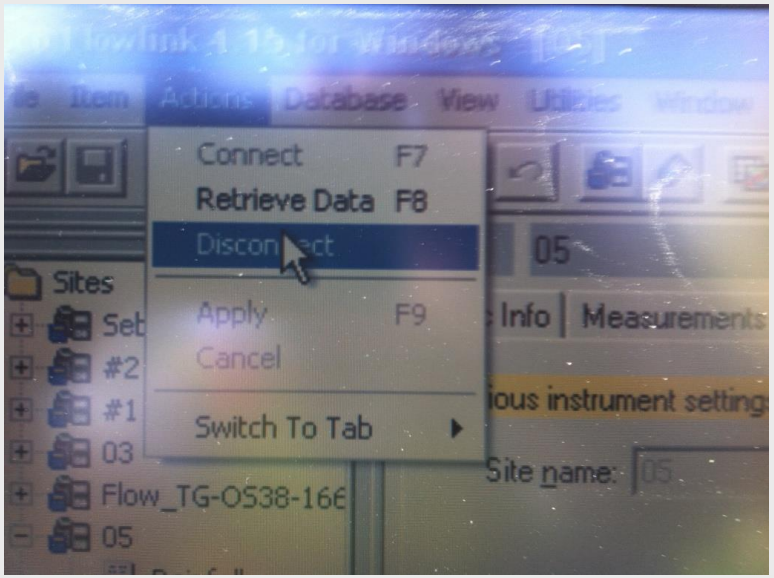
Procedures to collect data from the ISCO sampler



- Once the data is exported and saved now under the Actions tab the user needs to click disconnect.
- After disconnected then Flowlink program can be closed and the cables can be disconnected as well.
- After this it may be a good idea to turn off the ISCO unit for either replacing the battery or to lift the center section to check the bottles for collected samples.

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Visual Inspection of Sample Bottles

- To remove the center section flip up the latches put center section next to the tub section.
- Now visually verify to see if any samples have been collected in the bottles.
- Compare to the Site Setup Report to see if the report coincides with what was actually sampled.

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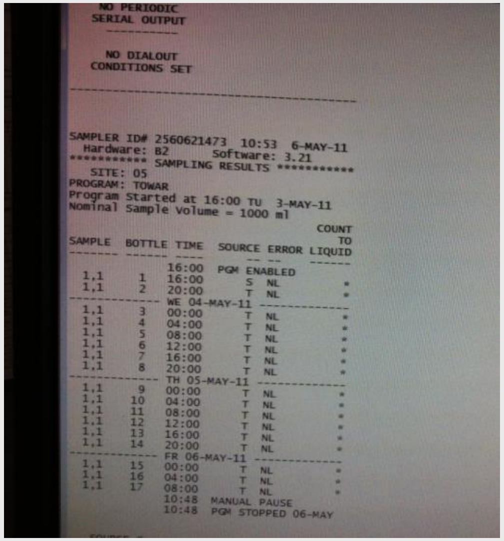
Compare Reports with actual Samples Collected



NO PERIODIC
SERIAL OUTPUT

NO DIALOUT
CONDITIONS SET

SAMPLER ID# 2560621473 10:53 6-MAY-11
Hardware: B2 Software: 3.21
*********** SAMPLING RESULTS ***********
SITE: 05
PROGRAM: TOWAR
Program Started at 16:00 TU 3-MAY-11
Nominal Sample Volume = 1000 ml

SAMPLE	BOTTLE	TIME	SOURCE	ERROR	COUNT TO LIQUID
		16:00	PGM ENABLED		
1,1	1	16:00	S	NL	*
1,1	2	20:00	T	NL	*
		WE 04-MAY-11			
1,1	3	00:00	T	NL	*
1,1	4	04:00	T	NL	*
1,1	5	08:00	T	NL	*
1,1	6	12:00	T	NL	*
1,1	7	16:00	T	NL	*
1,1	8	20:00	T	NL	*
		TH 05-MAY-11			
1,1	9	00:00	T	NL	*
1,1	10	04:00	T	NL	*
1,1	11	08:00	T	NL	*
1,1	12	12:00	T	NL	*
1,1	13	16:00	T	NL	*
1,1	14	20:00	T	NL	*
		FR 06-MAY-11			
1,1	15	00:00	T	NL	*
1,1	16	04:00	T	NL	*
1,1	17	08:00	T	NL	*
		10:48	MANUAL PAUSE		
		10:48	PGM STOPPED 06-MAY		

- After downloading the Site Setup Report the data should be looked at to see if the sampler collected any samples and is running properly.
- As shown above in this sample report NL stands for No Liquid.
- A majority of the time the user will either see a number under Count To Liquid or under error NM which stands for No More Liquid.
- In this case the water elevation was too low to collect samples so both the reports and the visual inspection coincide.

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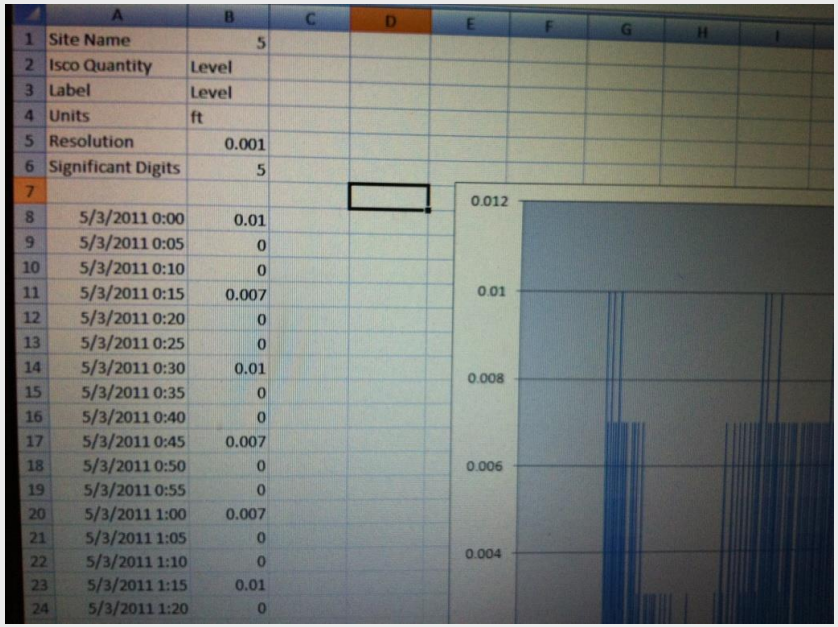
Compare downloaded level data to manual measured level



	A	B
1	Site Name	5
2	Isco Quantity	Level
3	Label	Level
4	Units	ft
5	Resolution	0.001
6	Significant Digits	5
7		
8	5/3/2011 0:00	0.01
9	5/3/2011 0:05	0
10	5/3/2011 0:10	0
11	5/3/2011 0:15	0.007
12	5/3/2011 0:20	0
13	5/3/2011 0:25	0
14	5/3/2011 0:30	0.01
15	5/3/2011 0:35	0
16	5/3/2011 0:40	0
17	5/3/2011 0:45	0.007
18	5/3/2011 0:50	0
19	5/3/2011 0:55	0
20	5/3/2011 1:00	0.007
21	5/3/2011 1:05	0
22	5/3/2011 1:10	0
23	5/3/2011 1:15	0.01
24	5/3/2011 1:20	0

- With the bubbler data shown above this is where the measurement of the water surface elevation to the bubbler tip can be compared.
- If the last recorded measurement is within 0.005 ft of the manually measured level then the device is working within the specifications and does not need to be calibrated.
- In this case the end of the bubbler tube is above the water elevation so the bubbler level should be 0.00’

Collecting of Sample Bottles

- In most cases the sampler will distribute liquid to the bottles and they will need to be capped and then taken for testing.
- A majority of the time the Turbidity test is taken right away
- If there is a significant rain event then other tests may be taken depending on what the Project Manager would like monitor.
- Run tests for parameters with low hold times as soon as possible.

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Replacing the Battery

- Once the bottles have been capped and collected the battery needs to be swapped out with a fully charged battery.
- The battery that has been in use needs to be put on charge once back in the office.

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Starting the ISCO Sampler Program to Run

- Once a fully charged battery has been installed the ISCO unit can be turned on to start sampling again.
- Start the program by selecting Run and then pressing enter . If any adjustments need to be made, instead of selecting Run the Program option needs to be selected.
- A majority of the time the only program option that changes is the time for the program to begin sampling.

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2. TOTAL SOLIDS

a. Testing Procedures

Testing Procedures – Total Solids

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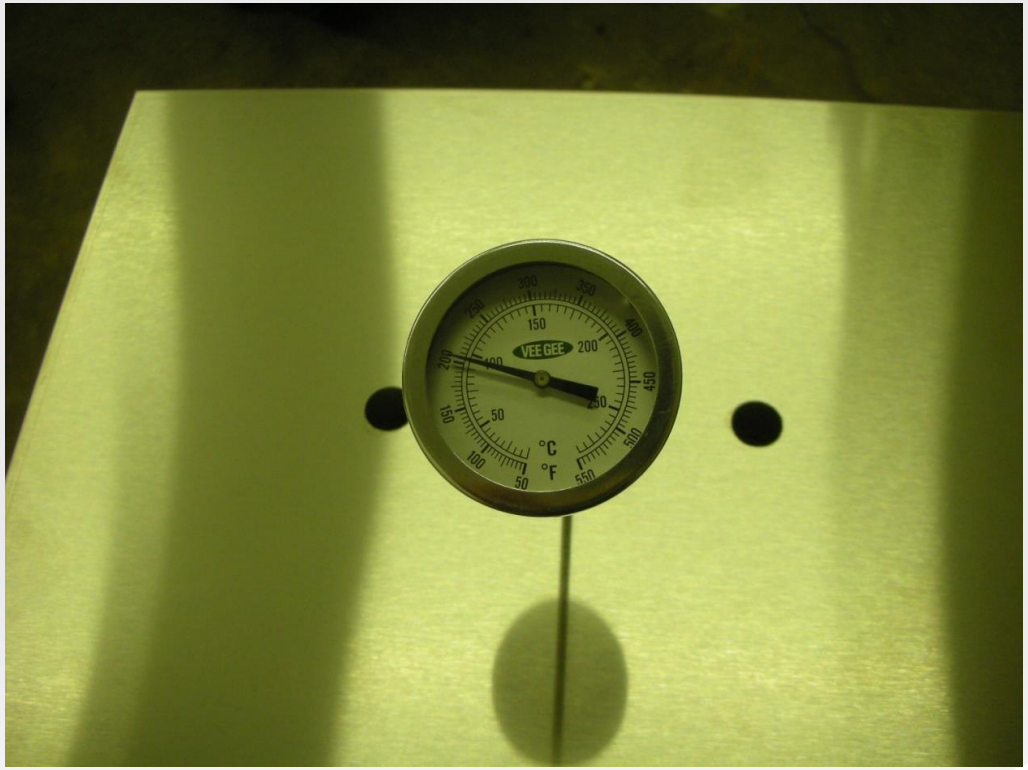
Testing Procedure for Total Solids

Oven Setting = 5.5 or 103°C-105°C

Testing Procedure for Total Solids



Oven Temperature = 103°C-105°C

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Testing Procedure for Total Solids

Turn on Scale to normalize for 1 hour

Testing Procedure for Total Solids

Prepare Desicator to receive empty dishes

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Testing Procedure for Total Solids

Dishes are found in this long box

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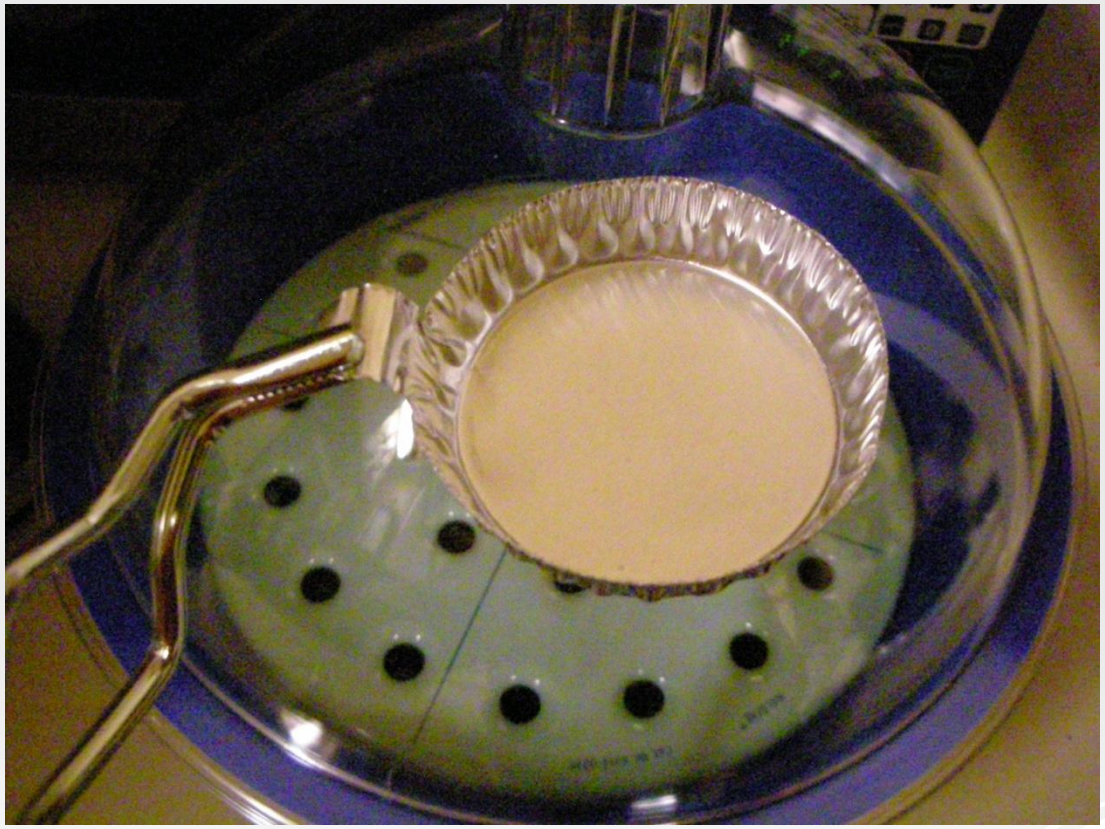
Testing Procedure for Total Solids



Do not touch trays with hands, tongs must be used

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Testing Procedure for Total Solids

Number each dish and place in desiccator

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Testing Procedure for Total Solids

Place trays in the oven for 2 hours

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Testing Procedure for Total Solids

Readings to be taken in mg

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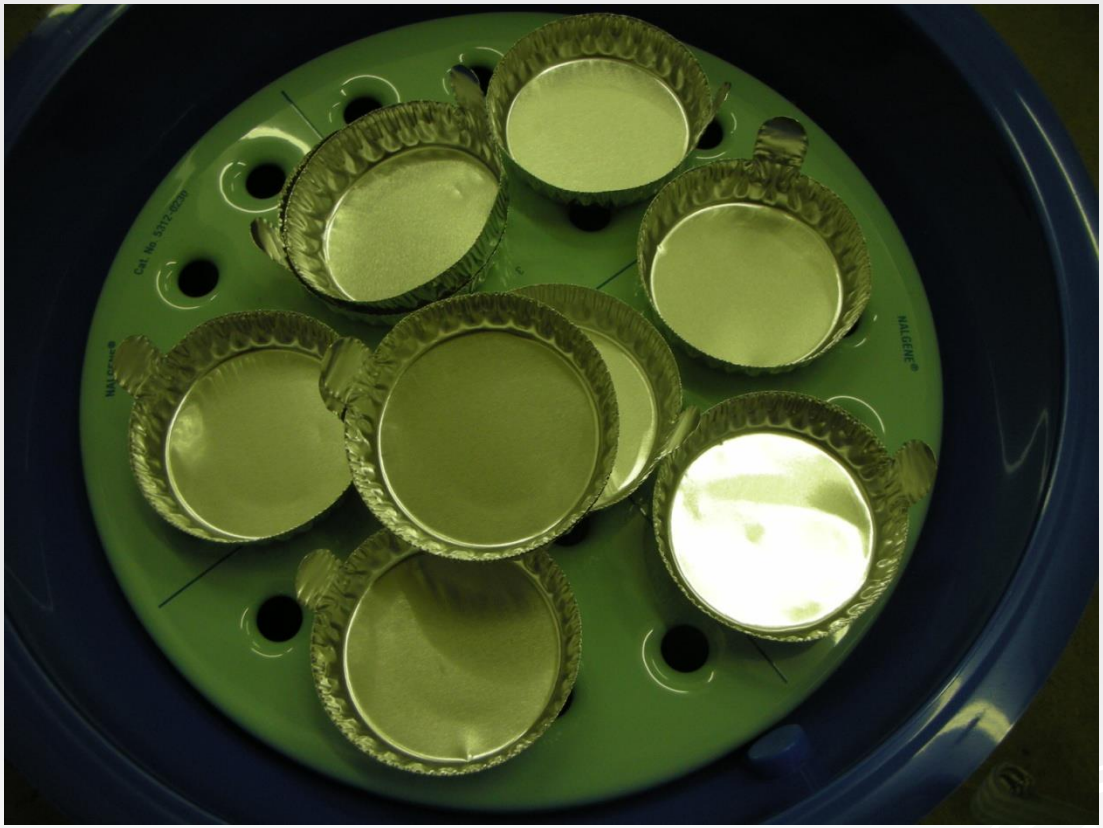
Testing Procedure for Total Solids



Remove trays from oven with tongs and place in desiccator

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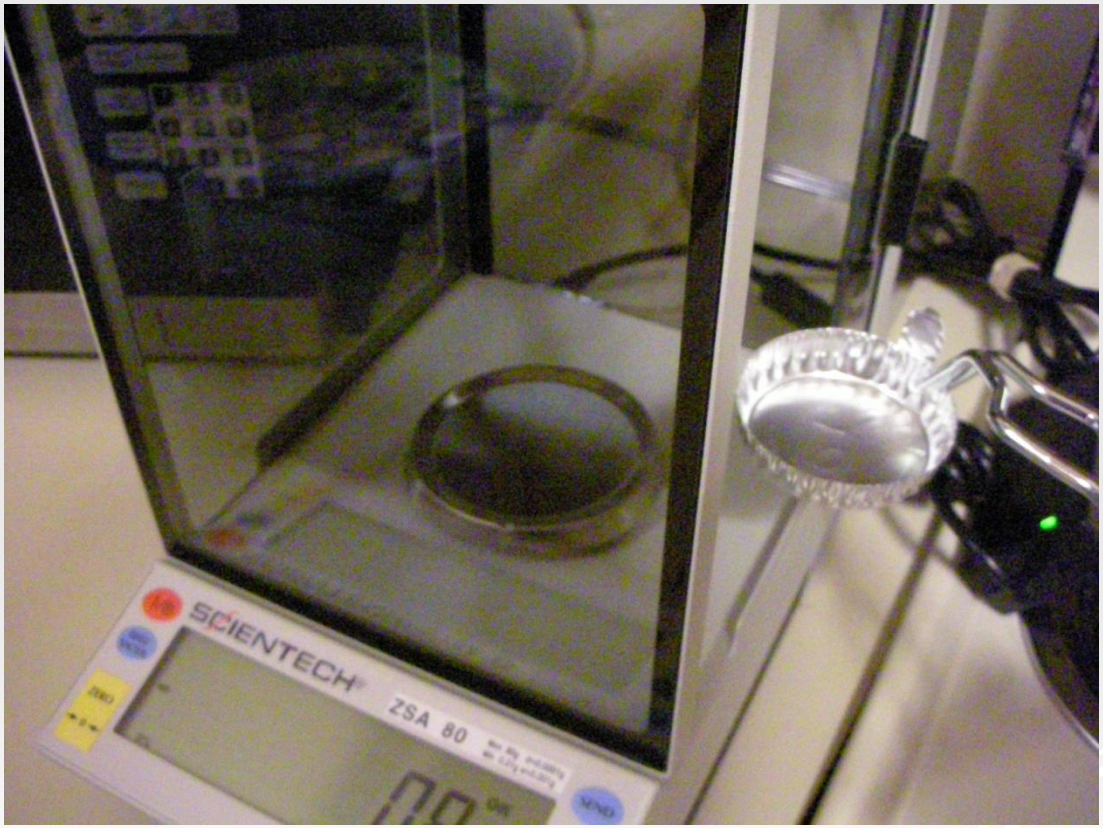
Testing Procedure for Total Solids



Zero scale and place tray in scale with tongs

Testing Procedure for Total Solids

Record mass of each dry tray (B)

Testing Procedure for Total Solids

Set pipette to 10 ml

Testing Procedure for Total Solids

Using 1-10 ml tensette pipette
Place 20 ml of sample in tray

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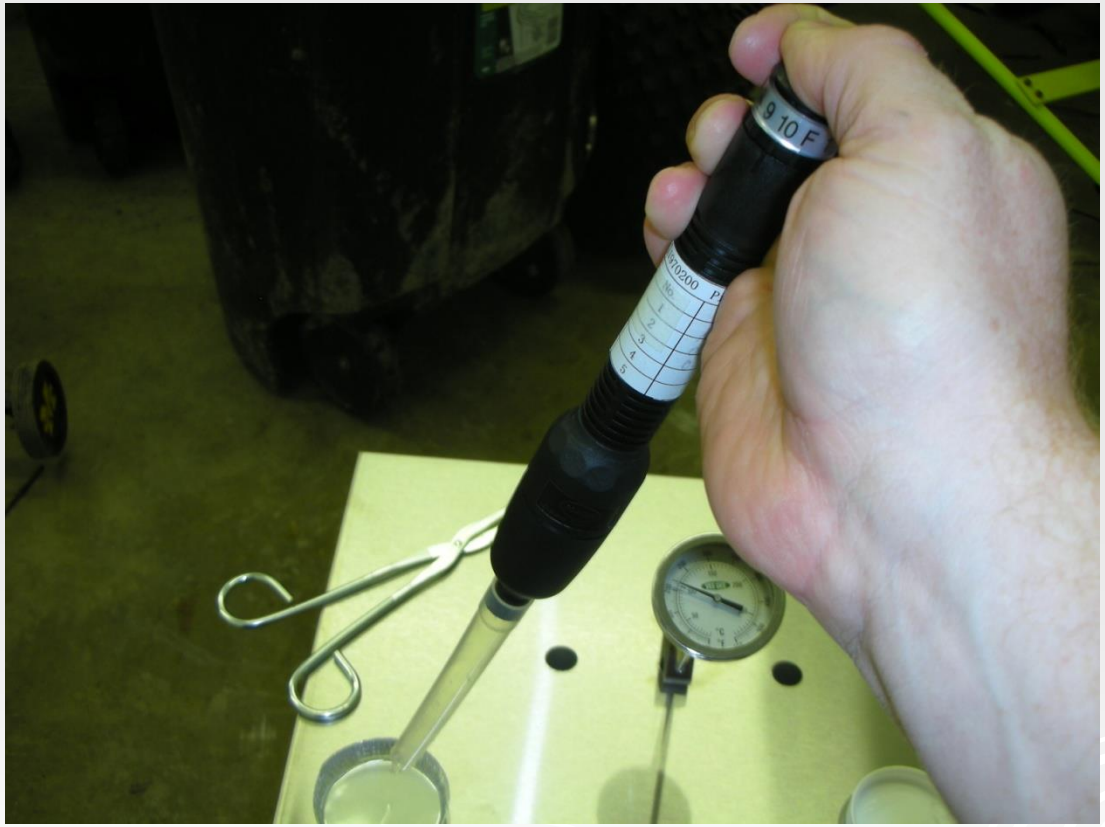
Testing Procedure for Total Solids



Place sample in tray

Testing Procedure for Total Solids

Place sample tray in oven at 103°C -105°C

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Testing Procedure for Total Solids

Samples to be dehydrated for 6 or more hours

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Testing Procedure for Total Solids

Remove dehydrated sample trays using tongs

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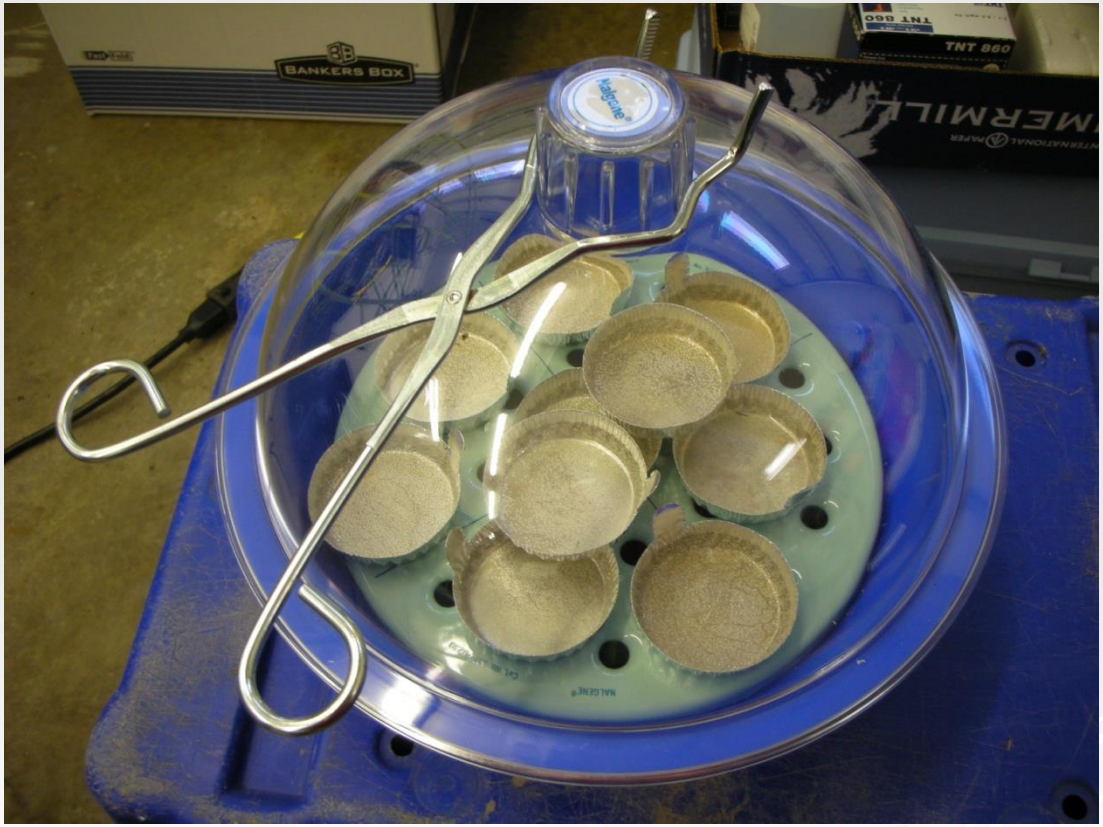
Testing Procedure for Total Solids

Place sample trays in desiccator to cool

Testing Procedure for Total Solids



Sample trays to be cooled and transported to scale

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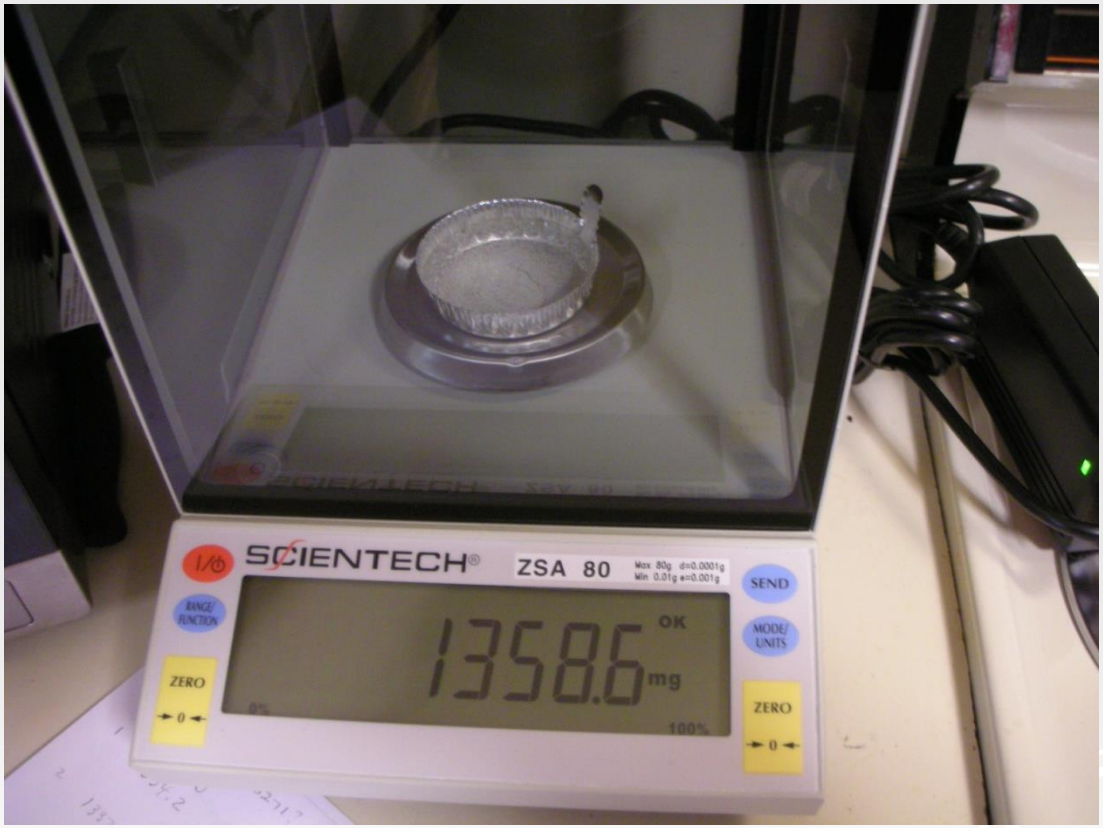
Testing Procedure for Total Solids



Record mass of sample trays (A)

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b. EPA Method 1684

EPA-821-R-01-015
January 2001

METHOD 1684

Total, Fixed, and Volatile Solids in Water, Solids, and Biosolids

Draft

January 2001

U.S. Environmental Protection Agency
Office of Water
Office of Science and Technology
Engineering and Analysis Division (4303)
1200 Pennsylvania Ave. NW
Washington, DC 20460

Acknowledgments

This method was prepared under the direction of William A. Telliard of the U.S. Environmental Protection Agency's (EPA's) Office of Water (OW), Engineering and Analysis Division (EAD). The method was prepared under EPA Contract 68-C-98-139 by DynCorp with assistance from Quality Works, Inc.

Disclaimer

This draft method has been reviewed and approved for publication by the Analytical Methods Staff within the Engineering and Analysis Division of the U.S. Environmental Protection Agency. Mention of trade names or commercial products does not constitute endorsement or recommendation for use. EPA plans further validation of this draft method. The method may be revised following validation to reflect results of the study. This method version contains minor editorial changes to the February 1999 version.

EPA welcomes suggestions for improvement of this method. Suggestions and questions concerning this method or its application should be addressed to:

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Requests for additional copies of this publication should be directed to:

Water Resource Center
Mail Code RC-4100
1200 Pennsylvania Ave., NW
Washington, DC 20460
(202) 260-7786 or (202) 260-2814

Note: This method is performance based. The laboratory is permitted to modify or omit any steps or procedure, provided that all performance requirements in this method are met. The laboratory may not omit any quality control analyses. The terms “shall”, “must”, and “may not” indicate steps and procedures required for producing reliable results. The terms “should” and “may” indicate optional steps that may be modified or omitted if the laboratory can demonstrate that the modified method produces results equivalent or superior to results produced by this method.

Method 1684

Total, Fixed, and Volatile Solids in Water, Solids, and Biosolids

1.0 Scope and Application

1.1 This method is applicable to the determination of total solids and the fixed and volatile fractions in such solid and semisolid samples as soils, sediments, biosolids (municipal sewage sludge), sludge separated from water and wastewater treatment processes, and sludge cakes from vacuum filtration, centrifugation, or other sludge dewatering processes.

1.2 This method is for use in the United States Environmental Protection Agency's (EPA's) data gathering and monitoring programs under the Clean Water Act, the Resource Conservation and Recovery Act, the Comprehensive Environmental Response, Compensation, and Liability Act, and the Safe Drinking Water Act.

1.3 Method detection limits (MDLs) and minimum levels (MLs) have not been formally established for this draft method. These values will be determined during the validation studies.

1.4 This method is performance based. The laboratory is permitted to omit any step or modify any procedure (e.g. to overcome interferences, to lower the cost of measurement), provided that all performance requirements in this method are met. Requirements for establishing method equivalency are given in Section 9.1.2.

1.5 Each laboratory that uses this method must demonstrate the ability to generate acceptable results using the procedure in Section 9.2.

2.0 Summary of Method

2.1 Sample aliquots of 25-50 g are dried at 103°C to 105°C to drive off water in the sample.

2.2 The residue from Section 2.1 is cooled, weighed, and dried again at 550°C to drive off volatile solids in the sample.

2.3 The total, fixed, and volatile solids are determined by comparing the mass of the sample before and after each drying step.

3.0 Definitions

Definitions for terms used in this method are given in the glossary at the end of the method (Section 18).

4.0 Interferences

4.1 Sampling, subsampling, and pipetting multi-phase samples may introduce serious errors. Make and keep such samples homogeneous during transfer. Use special handling to ensure sample integrity when subsampling. Mix small samples with a magnetic stirrer. If visible suspended

solids are present, pipette with wide-bore pipettes. If part of a sample adheres to the sample container, intensive homogenization is required to ensure accurate results. When dried, some samples form a crust that prevents evaporation; special handling such as extended drying times are required to deal with this. Avoid using a magnetic stirrer with samples containing magnetic particles.

4.2 The temperature and time of residue drying has an important bearing on results. Problems such as weight losses due to volatilization of organic matter, and evolution of gases from heat-induced chemical decomposition, weight gains due to oxidation, and confounding factors like mechanical occlusion of water and water of crystallization depend on temperature and time of heating. It is therefore essential that samples be dried at a uniform temperature, and for no longer than specified in the method. Each sample requires close attention to desiccation after drying. Minimize the time the desiccator is open because moist air may enter and be absorbed by the samples. Some samples may be stronger desiccants than those used in the desiccator and may take on water. If uptake of water by a sample is suspected, the operator should weigh the sample to see if it gains weight while in the desiccator. If the sludge is indeed taking on water, then a vacuum desiccator should be used.

4.3 Residues dried at 103°C to 105°C may retain some bound water as water of crystallization or as water occluded in the interstices of crystals. The residues also lose CO_2 in the conversion of bicarbonate to carbonate. The residues usually lose only slight amounts of organic matter by volatilization at this temperature. Because removal of occluded water is marginal at this temperature, attainment of constant weight may be very slow.

4.4 Results for residues high in oil or grease may be questionable because of the difficulty of drying to constant weight in a reasonable time.

4.5 The determination of both total and volatile solids is subject to negative error due to loss of ammonium carbonate and volatile organic matter during the drying step at 103°C to 105°C. Carefully observe specified drying time and temperature to control losses of volatile inorganic salts if these are a problem.

5.0 Safety

5.1 This method does not address all safety issues associated with its use. The toxicity or carcinogenicity of reagents used in this method have not been fully established. Each chemical and environmental sample should be regarded as a potential health hazard and exposure should be minimized. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses. Additional information on laboratory safety can be found in References 5-7.

5.2 All personnel handling environmental samples known to contain or to have been in contact with human waste should be immunized against known disease causative agents.

6.0 Equipment and Supplies

NOTE: *Brand names, suppliers, and part numbers are cited for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and materials other than those specified here, but demonstration of equivalent performance that meets the requirements of this method is the responsibility of the laboratory.*

6.1 Evaporating dishes–Dishes of 100-mL capacity. The dishes may be made of porcelain (90-mm diameter), platinum, or high-silica glass.

6.2 Watch glass–Capable of covering the evaporating dishes (Section 6.1).

6.3 Muffle furnace–Capable of maintaining a uniform temperature of 550°C throughout the drying chamber.

6.4 Steam bath for evaporation of liquid samples.

6.5 Desiccator–Moisture concentration in the desiccator should be monitored by an instrumental indicator or with a color-indicator desiccant.

6.6 Drying oven–Thermostatically-controlled, capable of maintaining a uniform temperature of 103°C to 105°C throughout the drying chamber.

6.7 Analytical balance–Capable of weighing to 0.1 mg for samples having a mass up to 200 g.

6.8 Reference weights–2 mg, 1000 mg, and 50g class "S" weights.

6.9 Container handling apparatus–Gloves, tongs, or a suitable holder for moving and handling hot containers after drying.

6.10 Sample handling apparatus–Spatulas, spoonulas, funnels, or other equipment for transfer and manipulation of sample.

6.11 Bottles–Glass or plastic bottles of a suitable size for sample collection.

6.12 Rubber gloves (Optional)

6.13 No. 7 Cork borer (Optional)

6.14 Dessicant (Optional)

7.0 Reagents and Standards

7.1 Reagent water–Deionized, distilled, or otherwise purified water.

7.2 Quality control spiking solution– If a commercially available standard can be purchased that contains standard fixed and volatile solids, the laboratory may use that standard. The laboratory may also prepare a spiking solution. One possible recipe is given below for a NaCl-KHP solution.

7.2.1 Dissolve 0.10 g sodium chloride (NaCl) in 500 mL reagent water. Mix to dissolve.

7.2.2 Add 0.10 g potassium hydrogen phthalate (KHP) to the NaCl solution (Section 7.2.1) and mix. If the KHP does not dissolve readily, warm the solution while mixing. Dilute to 1 L with reagent water. Store at 4°C. Assuming 100% volatility of the acid phthalate ion, this solution contains 200 mg/L total solids, 81.0 mg/L volatile solids, and 119 mg/L fixed solids.

8.0 Sample Collection, Preservation, and Storage

8.1 Use resistant-glass or plastic bottles to collect sample for solids analysis, provided that the material in suspension does not adhere to container walls. Sampling should be done in accordance with Reference 16.10. Begin analysis as soon as possible after collection because of the impracticality of preserving the sample. Refrigerate sample at 4°C up to the time of analysis to minimize microbiological decomposition of solids. Preferably do not hold samples more than 24 hours. Under no circumstances should the sample be held more than seven days. Bring samples to room temperature before analysis.

9.0 Quality Control

9.1 Each laboratory using this method is required to operate a formal quality control (QC) program. The minimum requirements of this program consist of an initial demonstration of laboratory capability, and the ongoing analysis of laboratory reagent blanks, precision and recovery standards, and matrix-spiked samples as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of data thus generated. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1.1 The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 9.2.

9.1.2 In recognition of advances that are occurring in analytical technology, the analyst is permitted certain options to improve separations or lower the costs of measurements, provided that all performance specifications are met. If an analytical technique other than the techniques specified in this method is used, that technique must have a specificity equal to or better than the specificity of the techniques in this method for total, fixed, and volatile solids in the sample of interest. Specificity is defined as producing results equivalent to the results produced by this method for laboratory-prepared solutions (Section 7.2) that meet all of the QC criteria stated in this method.

9.1.2.1 Each time a modification is made to this method, the analyst is required to repeat the Initial Precision and Recovery (IPR) test in Section 9.2.2 to demonstrate that the modification produces results equivalent to or better than results produced by this method. If the detection limit of the method will be affected by the modification, the analyst must demonstrate that the MDL (40 CFR part 136, appendix B) is less than or equal to the MDL in this method or one-third the regulatory compliance level, whichever is

higher. The tests required for this equivalency demonstration are given in Section 9.2.

9.1.2.2 The laboratory is required to maintain records of modifications made to this method. These records include the following, at a minimum:

9.1.2.2.1 The names, titles, addresses, and telephone numbers of the analyst(s) who performed the analyses and modification, and of the quality control officer who witnessed and will verify the analyses and modification.

9.1.2.2.2 A listing of pollutant(s) measured (total, fixed, and volatile solids).

9.1.2.2.3 A narrative stating reason(s) for the modification.

9.1.2.2.4 Results from all quality control (QC) tests comparing the modified method to this method, including:

(a) Initial precision and recovery (Section 9.2.2).
(b) Analysis of blanks (Section 9.3).
(c) Accuracy assessment (Section 9.5).
(d) Ongoing precision and recovery (Section 9.4).

9.1.2.2.5 Data that will allow an independent reviewer to validate each determination by tracing the instrument output (weight, absorbance, or other signal) to the final result. These data are to include:

(a) Sample numbers and other identifiers.
(b) Sample preparation dates.
(c) Analysis dates and times.
(d) Analysis sequence/run chronology.
(e) Sample weights.
(f) Make and model of analytical balance and weights traceable to NIST.
(g) Copies of logbooks, printer tapes, and other recordings of raw data.
(h) Data system outputs, and other data to link the raw data to the results reported.

9.1.3 Analyses of laboratory blanks are required to demonstrate freedom from contamination. The procedure and criteria for blank analyses are described in Section 9.3.

9.1.4 Analyses of ongoing precision and recovery (OPR) samples are required to demonstrate that the sample preparation and analysis are in control. The procedure and criteria for OPR samples are described in Section 9.4.

9.2 Initial demonstration of laboratory capability - The initial demonstration of laboratory capability is used to characterize laboratory performance and method detection limits.

9.2.1 Method detection limit (MDL) - The method detection limit must be established for the analyte, using the QC spiking solution (Section 7.2). To determine MDL values, take seven replicate aliquots of the diluted QC spiking solution and process each aliquot through each step of the analytical method. Perform all calculations and report the concentration values in the appropriate units. MDLs should be determined every year or whenever a modification to the method or analytical system is made that will affect the method detection limit.

9.2.2 Initial Precision and Recovery (IPR) - To establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations:

9.2.2.1 Prepare four samples by diluting the QC spiking solution (Section 7.2) to 1-5 times the ML. Using the procedures in Section 11, analyze these samples for total, fixed, and volatile solids.

9.2.2.2 Using the results of the four analyses, compute the average percent recovery (x) and the standard deviation (s, Equation 1) of the percent recovery for total, fixed, and volatile solids.

Equation 1

$$s = \sqrt{\frac{\sum x^2 - \frac{(\sum x^2)}{n}}{n-1}}$$

Where:

n = number of samples
x = % recovery in each sample
s = standard deviation

9.2.2.3 Compare s and x with the corresponding limits for initial precision and recovery in Table 1. If s and x meet the acceptance criteria, system performance is acceptable and analysis of samples may begin. If, however, s exceeds the precision limit or x falls outside the range for recovery, system performance is unacceptable. In this event, correct the problem, and repeat the test.

9.3 Laboratory blanks

9.3.1 Prepare and analyze a laboratory blank initially (i.e. with the tests in Section 9.2) and with each analytical batch. The blank must be subjected to the same procedural steps as a sample, and will consist of approximately 25 g of reagent water.

9.3.2 If material is detected in the blank at a concentration greater than the MDL (Section 1.3), analysis of samples must be halted until the source of contamination is eliminated and a new blank shows no evidence of contamination. All samples must be associated with an uncontaminated laboratory blank before the results may be reported for regulatory compliance purposes. Sample results are also acceptable for regulatory compliance purposes if

they are associated with a blank that contains less than 1/10 the concentration of the analyte(s) of interest in the associated samples.

9.4 Ongoing Precision and Recovery (OPR).

9.4.1 Prepare an OPR solution identical to the IPR solution described in Section 9.2.2.1.

9.4.2 An aliquot of the OPR solution must be analyzed with each preparation batch (samples of the same matrix started through the sample preparation process (Section 11) on the same 12-hour shift, to a maximum of 10 samples).

9.4.3 Compute the percent recovery of total, fixed, and volatile solids in the OPR sample.

9.4.4 For each analyte, compare the results to the limits for ongoing recovery in Table 1. If all analytes meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may proceed. If, however, the recovery of an analyte falls outside of the range given, the analytical processes are not being performed properly for that analyte. Correct the problem, reprepare the sample batch, and repeat the OPR test. All samples must be associated with an OPR analysis that passes acceptance criteria before the sample results can be reported for regulatory compliance purposes.

9.4.5 Add results that pass the specifications in Section 9.4.4 to IPR and previous OPR data. Update QC charts to form a graphic representation of continued laboratory performance. Develop a statement of laboratory accuracy for each analyte by calculating the average percent recovery (R) and the standard deviation of percent recovery (SR). Express the accuracy as a recovery interval from R-2SR to R+2SR. For example, if R=95% and SR=5%, the accuracy is 85-105%.

9.5 Duplicate analyses

9.5.1 Ten percent of samples must be analyzed in duplicate. The duplicate analyses must be performed within the same sample batch (samples whose analysis is started within the same 12-hour period).

9.5.2 The results of duplicate samples analyzed for total, fixed, and volatile solids must be within 10% of the solids determination.

10.0 Calibration and Standardization

10.1 Calibrate the analytical balance at 2 mg and 1000 mg using class "S" weights.

10.2 Calibration shall be within ± 10% (i.e. ±0.2 mg) at 2 mg and ± 0.5% (i.e. ±5 mg) at 1000 mg. If values are not within these limits, recalibrate the balance.

10.3 Place a 50 g weight and a 2 mg on the balance. Verify that the balance reads 50.002 ± 10 % (i.e. ± 0.2 mg).

11.0 Procedure

11.1 Total Solids

11.1.1 Preparation of evaporating dishes–If volatile solids are to be measured, ignite clean evaporating dishes and watch glasses at 550°C for 1 hour in a muffle furnace. If only total solids are to be measured, heat dishes and watch glasses at 103°C to 105°C for 1 hour in an oven. Cool and store the dried equipment in a desiccator. Weigh each dish and watch glass prior to use (record combined weight as "W_{dish}").

11.1.2 Preparation of samples.

11.1.2.1 Fluid samples–If the sample contains enough moisture to flow readily, stir to homogenize, place a 25 to 50 g sample aliquot on a prepared evaporating dish. If the sample is to be analyzed in duplicate, the mass of the two aliquots may not differ by more than 10%. Cover each sample with a watch glass, and weigh to the nearest 0.01 g (record weight as "W_{sample}"). Spread each sample so that it is evenly distributed over the evaporating dish. Evaporate the samples to dryness on a steam bath.

NOTE: *Weigh wet samples quickly because wet samples tend to lose weight by evaporation. Samples should be weighed immediately after aliquots are prepared.*

11.1.2.2 Solid samples–If the sample consists of discrete pieces of solid material (dewatered sludge, for example), take cores from each piece with a No. 7 cork borer or pulverize the entire sample coarsely on a clean surface by hand, using rubber gloves. Place a 25 to 50 g aliquot of the pulverized sample on a prepared evaporating dish. If the sample is to be analyzed in duplicate, the mass of the two aliquots may not differ by more than 10%. Cover each sample with a watch glass, and weigh (record weight as "W_{sample}"). Spread each sample so that it is evenly distributed over the evaporating dish.

11.1.3 Dry the samples at 103°C to 105°C for 12 hours, minimum, cool to balance temperature in an individual desiccator containing fresh desiccant, and weigh.

NOTE: *It is imperative that dried samples be weighed quickly since residues often are very hygroscopic and rapidly absorb moisture from the air. Samples must remain in the dessicator until the analyst is ready to weigh them.*

11.1.4 Heat the residue for 1 hour, cool it to balance temperature in a desiccator, and weigh. Repeat this heating, cooling, desiccating, and weighing procedure until the weight change is less than 4% or 50 mg, whichever is less. Record the final weight as "W_{total}."

11.2 Fixed and volatile solids.

11.2.1 Transfer the evaporating dishes containing the dried residues (Section 11.1.4) to a cool muffle furnace. Heat the furnace to 550°C and ignite it for 2 hours.

NOTE: *If the residue contains large amounts of organic matter, first ignite it over a gas burner and under an exhaust hood in the presence of adequate air to lessen losses due to reducing conditions and to avoid odors in the laboratory.*

11.2.2 Cool the residue in a desiccator to balance the temperature. Weigh the residues. Repeat igniting (30 min), cooling, desiccating, and weighing steps until the weight change is less than 4% or 50 mg, whichever is less. Record the final weight as "$W_{volatile}$."

12.0 Data Analysis and Calculations

12.1 Calculate the % solids or the mg solids/kg sludge for total solids (Equation 2), fixed solids, (Equation 3), and volatile solids (Equation 4).

Equation 2

$$\%\ total\ solids = \frac{W_{total} - W_{dish}}{W_{sample} - W_{dish}} * 100$$

or

$$\frac{mg\ total\ solids}{kg\ sludge} = \frac{W_{total} - W_{dish}}{W_{sample} - W_{dish}} * 1{,}000{,}000$$

Where:

W_{dish} = *Weight of dish (mg)*
W_{sample} = *Weight of wet sample and dish (mg)*
W_{total} = *Weight of dried residue and dish (mg)*

Equation 3

$$\%\ fixed\ solids = \frac{W_{volatile} - W_{dish}}{W_{total} - W_{dish}} * 100$$

or

$$\frac{mg\ fixed\ solids}{kg\ sludge} = \frac{W_{volatile} - W_{dish}}{W_{total} - W_{dish}} * 1{,}000{,}000$$

Where:

W_{dish} = *Weight of dish (mg)*
W_{total} = *Weight of dried residue and dish (mg)*
$W_{volatile}$ = *Weight of residue and dish after ignition (mg)*

Equation 4

$$\%\ volatile\ solids = \frac{W_{total} - W_{volatile}}{W_{total} - W_{dish}} * 100$$

or

$$\frac{mg\ volatile\ solids}{kg\ sludge} = \frac{W_{total} - W_{volatile}}{W_{total} - W_{dish}} * 1{,}000{,}000$$

Where:

W_{dish} = *Weight of dish (mg)*
W_{total} = *Weight of dried residue and dish (mg)*
$W_{volatile}$ = *Weight of residue and dish after ignition (mg)*

12.2 Sample results should be reported as % solids or mg/kg to three significant figures. Report results below the ML as < ML, or as required by the permitting authority or in the permit. Duplicate determinations must agree within 10% of their average.

13.0 Method Performance

13.1 Method performance (MDL and quality control acceptance criteria) will be determined during the multi-lab validation of this method.

13.2 Total, fixed, and volatile solids duplicate determinations must agree within 10% to be reported for permitting purposes. If duplicate samples do not meet this criteria, the problem must be discovered and the sample must be run over.

14.0 Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The Environmental Protection Agency has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction", available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington D.C. 20036, (202)872-4477.

15.0 Waste Management

15.1 The Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench

operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult "The Waste Management Manual for Laboratory Personnel", available from the American Chemical Society at the address listed in the Section 14.2.

16.0 References

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17.0 Tables, Diagrams, Flowcharts, and Validation Data

17.1 Table 1 - Quality Control Acceptance Criteria for Method 1684.

18.0 Definitions

18.1 Analytical batch–The set of samples analyzed at the same time, to a maximum of 10 samples. Each analytical batch of 10 or fewer samples must be accompanied by a laboratory blank (Section 9.3), an ongoing precision and recovery sample (OPR, Section 9.6), and a set of duplicate samples, resulting in a minimum of five analyses (1 sample, 1 blank, 1 OPR, 2 duplicates) and a maximum of 14 analyses.

18.2 Fixed solids–The residue left in the vessel after a sample is ignited (heated to dryness at 550°C).

18.3 Initial precision and recovery (IPR)–Four aliquots of the diluted PAR analyzed to establish the ability to generate acceptable precision and accuracy. An IPR is performed the first time this method is used and any time the method or instrumentation is modified.

18.4 IPR–See initial precision and recovery.

18.5 Laboratory blank (method blank)–An aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment and reagents that are used with samples. The laboratory blank is used to determine if analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.

18.6 Laboratory control sample (LCS)–See Ongoing precision and recovery standard (OPR).

18.7 May–This action, activity, or procedural step is neither required nor prohibited.

18.8 May not–This action, activity, or procedural step is prohibited.

18.9 Method detection limit (MDL)–The lowest level at which an analyte can be detected with 99 % confidence that the analyte concentration is greater than zero.

18.10 Must–This action, activity, or procedural step is required.

18.11 Ongoing precision and recovery standard (OPR, also called a laboratory control sample)–A laboratory blank spiked with known quantities of analytes. The OPR is analyzed exactly like a sample. Its purpose is to assure that the results produced by the laboratory remain within the limits specified in this method for precision and accuracy.

18.12 OPR–See Ongoing precision and recovery standard.

18.13 PAR–See Precision and recovery standard.

18.14 Precision and recovery standard–Secondary standard that is diluted and spiked to form the IPR and OPR.

18.15 Quality control sample (QCS):–A sediment sample containing analytes of interest at known concentrations. The QCS is obtained from a source external to the laboratory or is prepared from standards obtained from a different source than the calibration standards. The purpose is to check laboratory performance using test materials that have been prepared independently from the normal preparation process.

18.16 Reagent Water– Water that should be free of substances that interfere with analytical methods.

18.17 Sediment sample–A fluvial, sand and/or humic sample matrix exposed to a marine, brackish or fresh water environment. It is limited by this method to that portion which may be passed through a number 10 sieve or a 2 mm mesh sieve.

18.18 Shall–This action, activity or procedural step is required.

18.19 Should–This action, activity, or procedural step is suggested but not required.

18.20 Total solids–The residue left in the vessel after evaporation of liquid from a sample and subsequent drying in an oven at 103°C to 105°C.

18.21 Volatile solids–The weight loss after a sample is ignited (heated to dryness at 550°C). Determinations of fixed and volatile solids do not distinguish precisely between inorganic and organic matter because the loss on ignition is not confined to organic matter. It includes losses due to decomposition or volatilization of some mineral salts.

Table 1 - Quality Control Acceptance Criteria for Method 1684[1]

Analyte	MDL	IPR		OPR
		x	s	x
total solids	3 mg/L	85-110%	10% Rsd	80-110%
fixed solids	7 mg/L	75-110%	20% Rsd	70-110%
volatile solids	7 mg/L	75-110%	30% Rsd	70-110%

[1] Performance criteria are initial estimates. These estimates serve as data quality objectives for the single laboratory validation of this method.

3. TOTAL SUSPENDED SOLIDS

a. Testing Procedures

Testing Procedures – Total Suspended Solids

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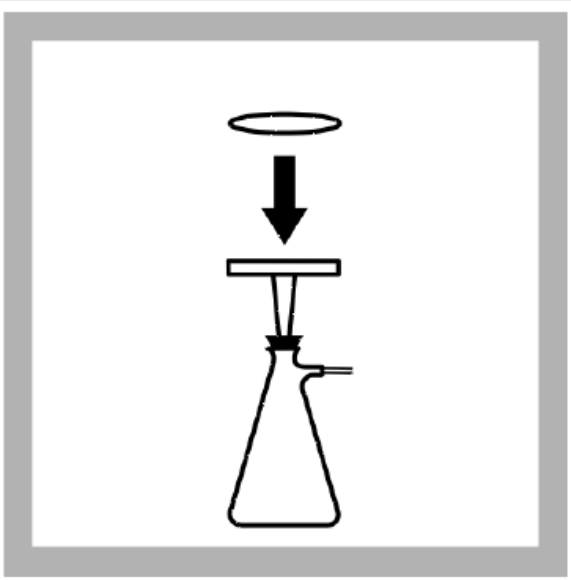
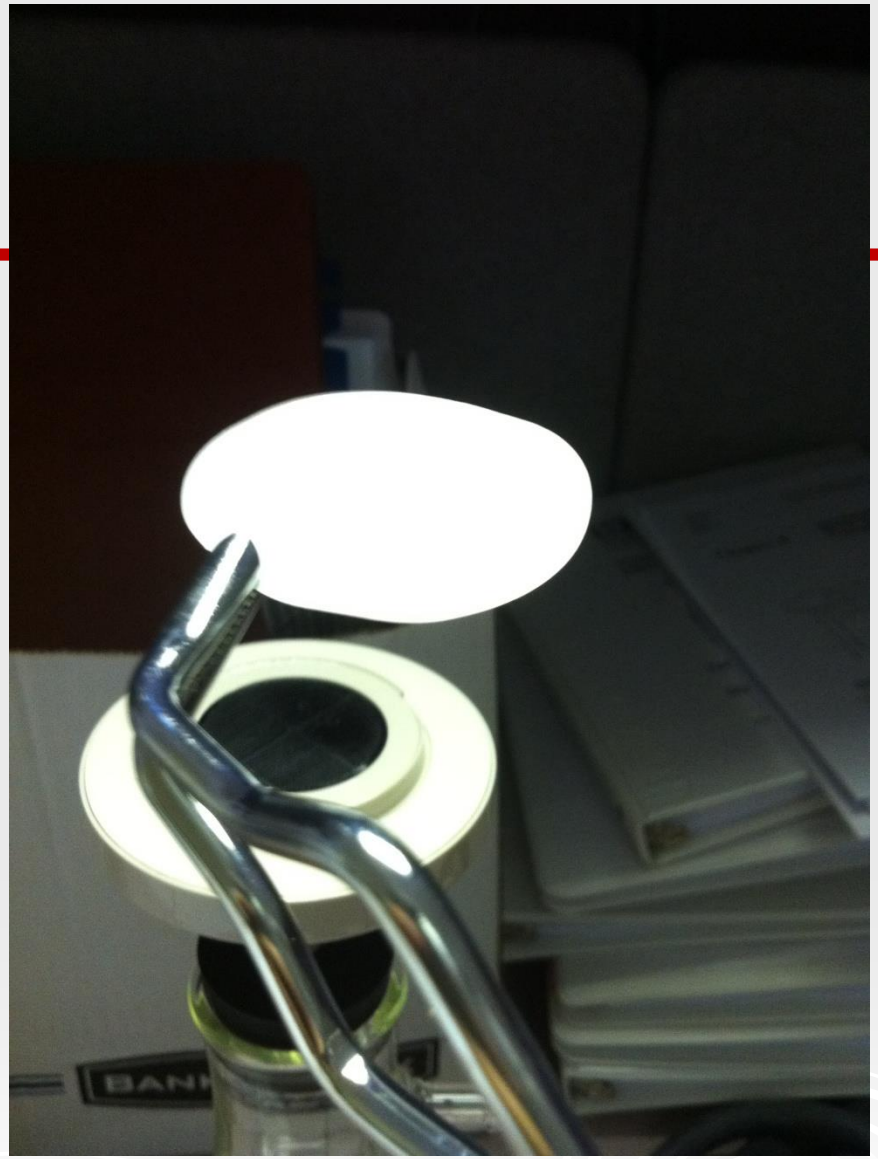


1. Use tweezers to place a 47-mm glass fibre filter disc in the filter holder. Always use tweezers to handle filter discs.

Moisture from fingers can add moisture to the disc and cause a weighing error.



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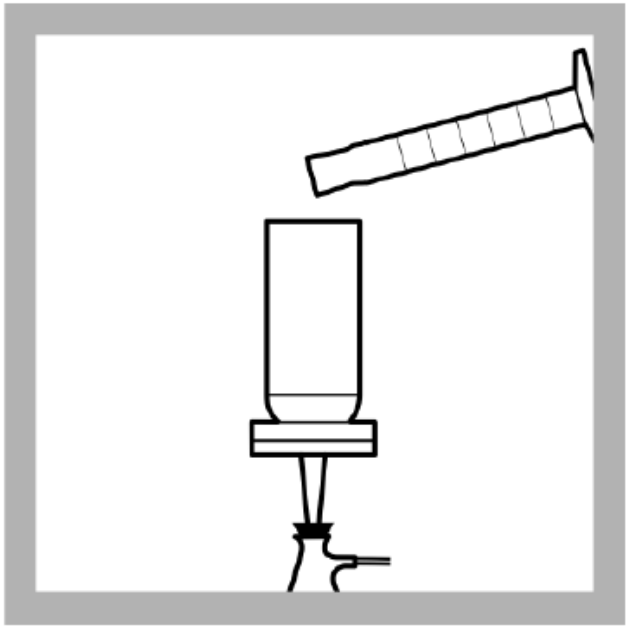

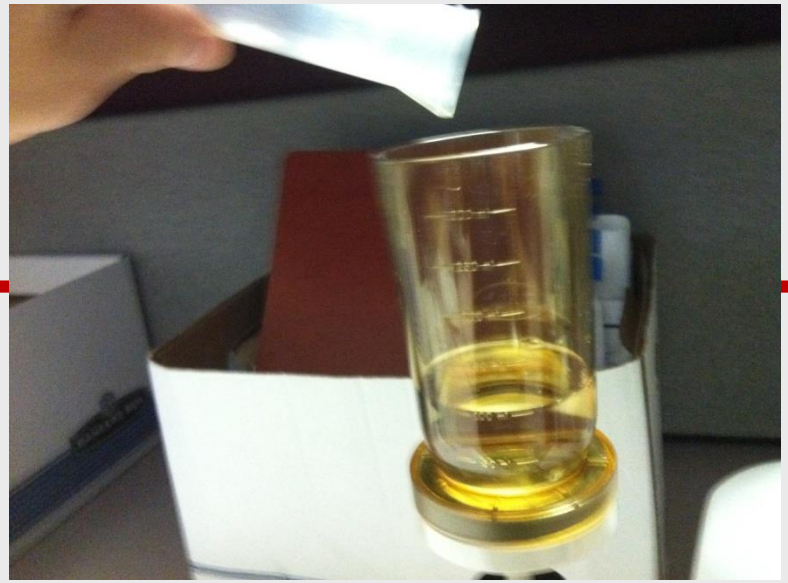

2. Place the filter holder assembly in the filtering flask and add 100 mL of deionized water. Apply vacuum to the flask until all the water is drawn through the filter.

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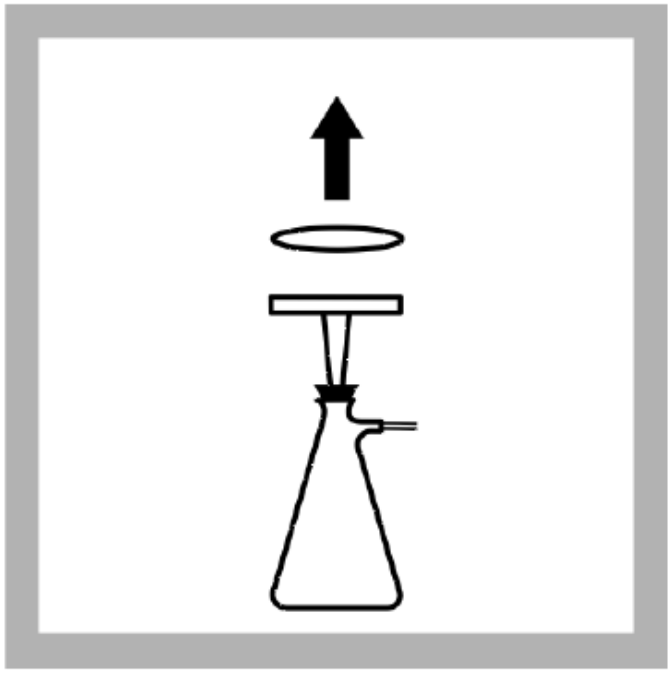

3. Slowly release the vacuum from the filtering system and remove the disc from the filter holder and transfer to a watch glass.

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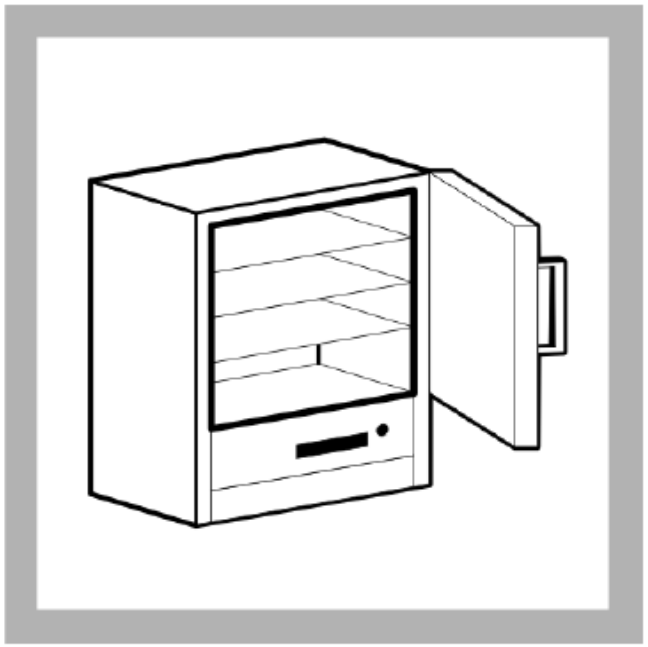
Testing Procedure for Total Suspended Solids



4. Place the disc in a preheated drying oven at 103 °C for one hour.

Oven Setting = 5.5 or 103°C-105°C

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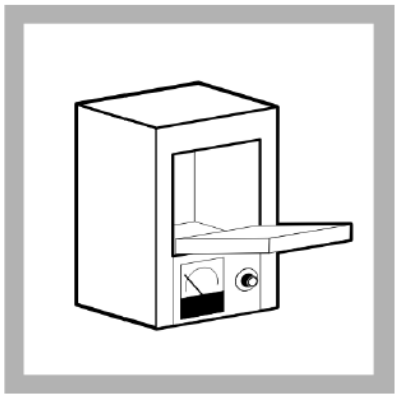

5. If volatile nonfilterable solids are also being measured, use tongs to place the watch glass with the disc into a muffle furnace and ignite at 550 °C for 15 minutes. If not, omit this step.

Partially preheat the muffle furnace before inserting the watch glass. Placing the watch glass in a 550 °C furnace could cause it to shatter. Bring the temperature up to 550 °C 15 minutes after placing the filter and watch glass in the furnace.

6. Use metal tongs to remove the disc and watch glass from the oven or furnace and place in a desiccator. Cover immediately. Allow the watch glass to cool slightly before sealing the desiccator as pressure from the heated air inside the desiccator can force the cover off.

Allow the filter and glass to cool to room temperature.

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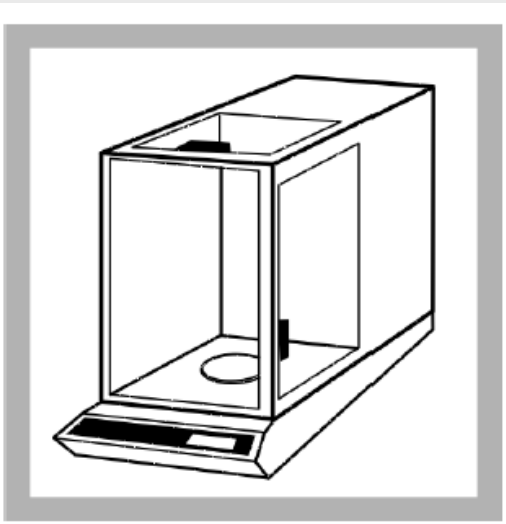

7. Remove the watch glass and disc from the desiccator as a unit and place beside the analytical balance.

Use plastic tweezers to remove the disc from the watch glass and weigh to the nearest 0.1 mg (0.0001 g). Record this value as B.

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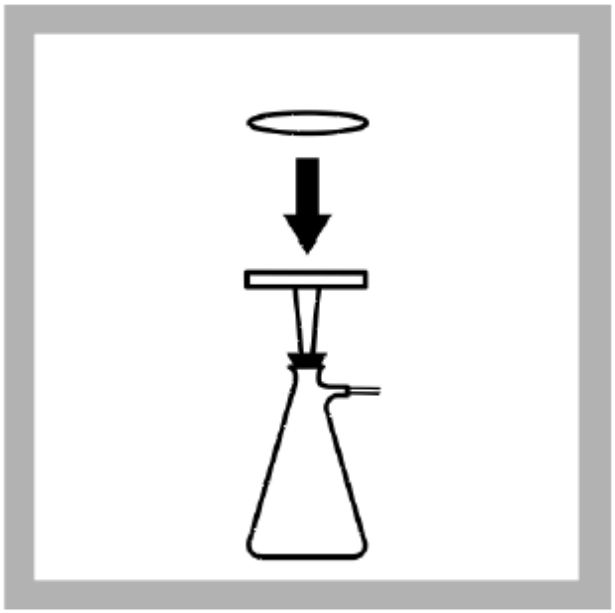

8. Again, place the disc in the filter holder/flask assembly. Wet the disc with deionized water to ensure adhesion to the holder.

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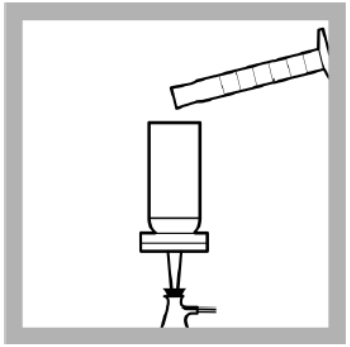
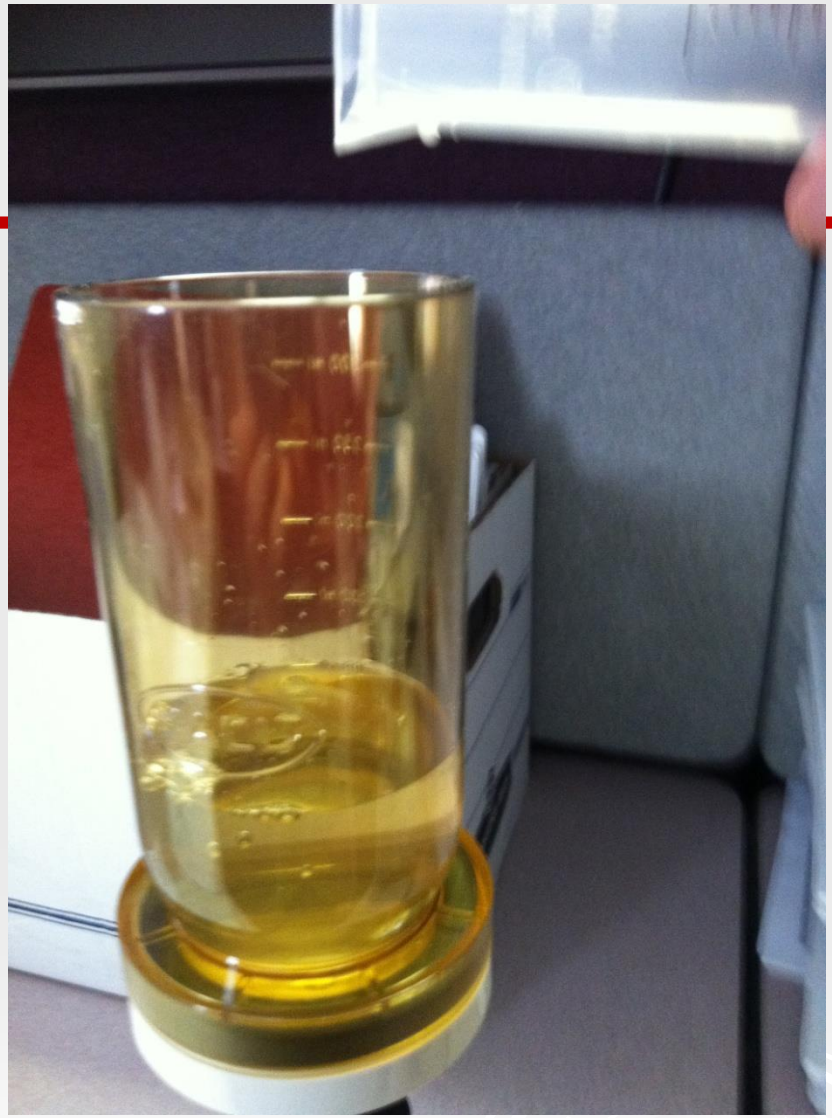


9. Filter 100 mL (or more, if solids content is low) of well-mixed, representative water sample by applying vacuum to the flask. Follow with three separate 10-mL washings of deionized water.

For greatest accuracy, filter as much sample as possible. However, using a sample that contains more than 15 mg of solids will clog the filter prematurely. Adjust the exact volume of the water sample to achieve the optimum condition. Several completed tests will show whether any adjustment is necessary.



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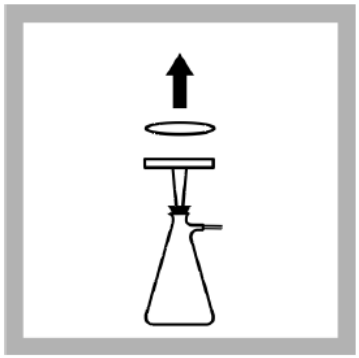
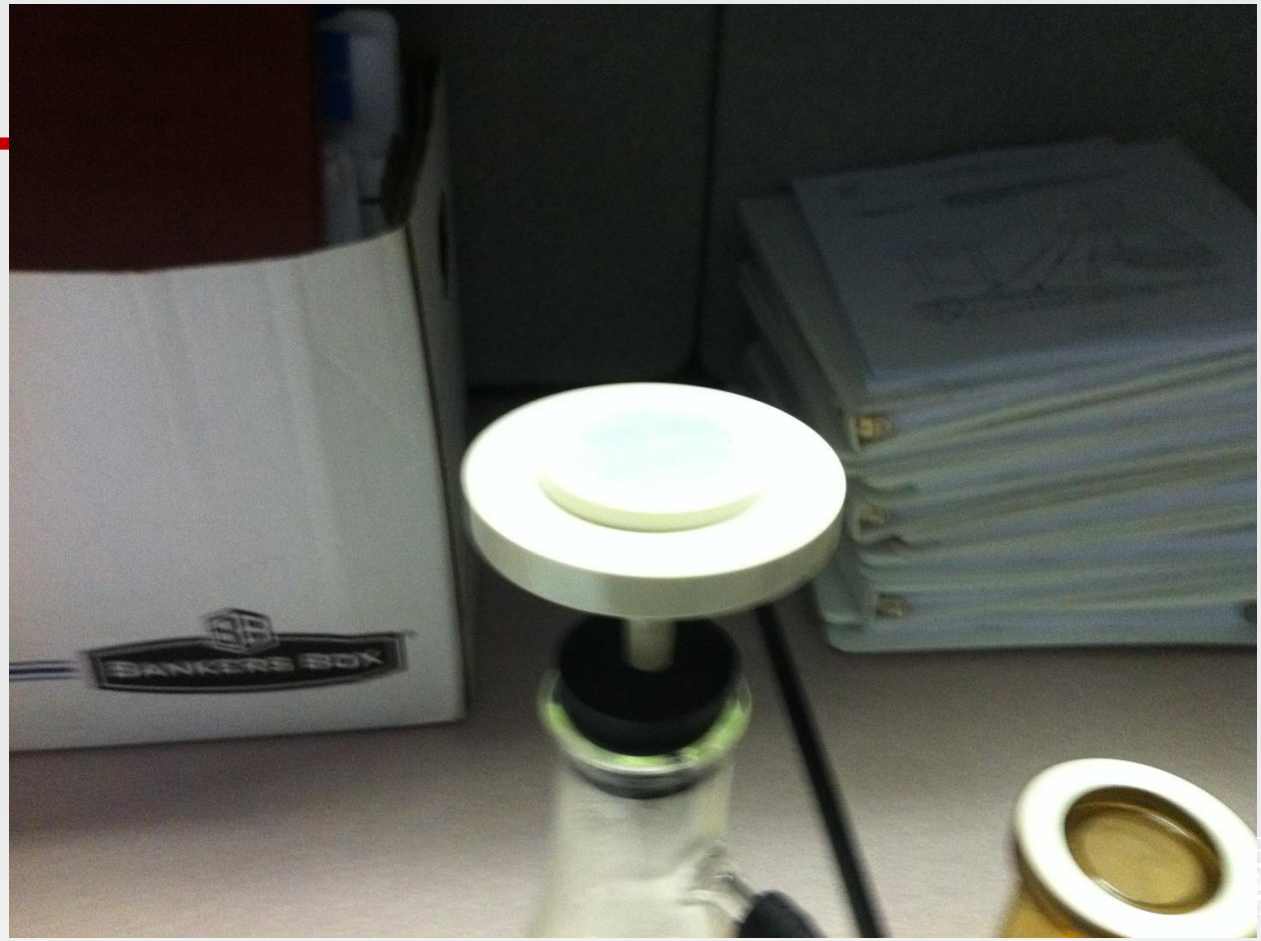


10. Remove any residue that remains on the sides or bottom lip of the filter holder. A rubber policeman on the end of a stirring rod is very helpful to scrape the residue loose. Small amounts of deionized water will help wash the residue down onto the filter disc.

Slowly release the vacuum from the filtering system and gently remove the filter disc from the holder. Place the disc on a watch glass. Inspect the filtrate (filtered water in flask) to make sure that the solids are properly trapped on the disc.



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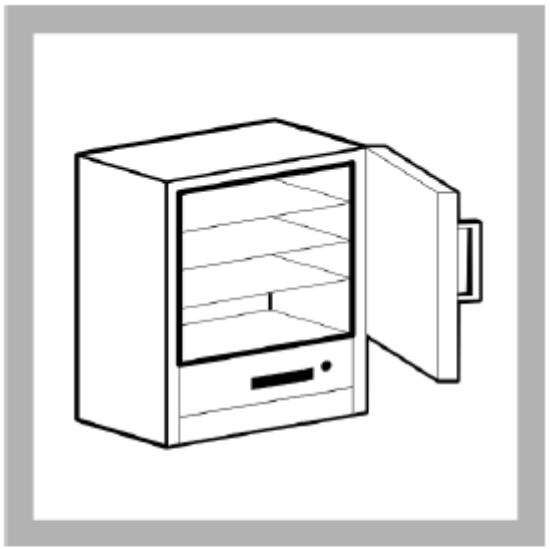

11. Place the watch glass and filter in a drying oven at 103 °C for one hour.

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12. Use metal tongs to remove the disc and watch glass from the oven or furnace and place in a desiccator. Cover immediately. Allow the watch glass to cool slightly before sealing the desiccator as pressure from the heated air inside the desiccator can force the cover off.

Allow the filter and glass to cool to room temperature.

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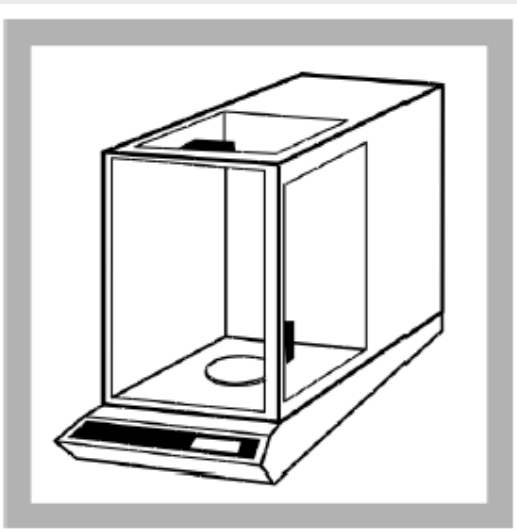

13. Remove the watch glass and disc from the desiccator as a unit and place beside the analytical balance.

Use plastic tweezers to remove the disc from the watch glass and weigh to the nearest 0.1 mg (0.0001 g). Record this mg value as A.

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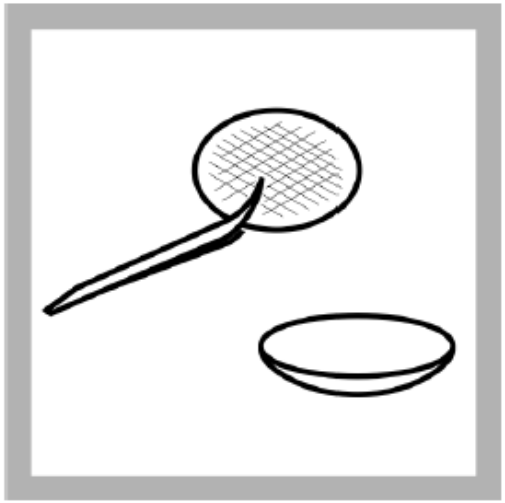

14. Return the disc to the watch glass if the mg/L Volatile Nonfilterable Residue is to be determined. If not, discard the disc.

If Volatile Nonfilterable Residue is to be determined, do not lose any of the suspended matter on the disc.

15. Calculate Total Non-filterable Residue (TNR):

$$\frac{A - B}{\text{Sample Volume in Liters}} = \text{mg/L TNR}$$

Where:

A = Weight (mg) of disc with residue

B = Weight (mg) of disc

Example:

A = 95.5 mg

B = 81.5 mg

Volume of sample = 0.1 L

$$\frac{95.5\ \text{mg} - 81.5\ \text{mg}}{0.1\ \text{L}} = 140\ \text{mg/L TNR}$$

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b. EPA Method 160.2

METHOD #: 160.2	Approved for NPDES (Issued 1971)
TITLE:	Residue, Non-Filterable (Gravimetric, Dried at 103-105°C)
ANALYTE:	Residue ,Non-Filterable
INSTRUMENTATION:	Drying Oven
STORET No.	00530

1.0 Scope and Application

1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
1.2 The practical range of the determination is 4 mg/L to 20,000 mg/L.

2.0 Summary of Method

2.1 A well-mixed sample is filtered through a glass fiber filter, and the residue retained on the filter is dried to constant weight at 103-105°C.
2.2 The filtrate from this method may be used for Residue, Filterable.

3.0 Definitions

3.1 Residue, non-filterable, is defined as those solids which are retained by a glass fiber filter and dried to constant weight at 103-105°C.

4.0 Sample Handling and Preservation

4.1 Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result.
4.2 Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to 4°C, to minimize microbiological decomposition of solids, is recommended.

5.0 Interferences

5.1 Filtration apparatus, filter material, pre-washing, post-washing, and drying temperature are specified because these variables have been shown to affect the results.
5.2 Samples high in Filterable Residue (dissolved solids), such as saline waters, brines and some wastes, may be subject to a positive interference. Care must be taken in selecting the filtering apparatus so that washing of the filter and any dissolved solids in the filter (7.5) minimizes this potential interference.

6.0 Apparatus

6.1 Glass fiber filter discs, without organic binder, such as Millipore AP-40, Reeves Angel 934-AH, Gelman type A/E, or equivalent.
NOTE: Because of the physical nature of glass fiber filters, the absolute pore size cannot be controlled or measured. Terms such as "pore size", collection efficiencies and effective retention are used to define this property in glass fiber filters. Values for these parameters vary for the filters listed above.

6.2 Filter support: filtering apparatus with reservoir and a coarse (40-60 microns) fritted disc as a filter support.
NOTE: Many funnel designs are available in glass or porcelain. Some of the most common are Hirsch or Buchner funnels, membrane filter holders and Gooch crucibles. All are available with coarse fritted disc.

6.3 Suction flask.

6.4 Drying oven, 103-105°C.

6.5 Desiccator.

6.6 Analytical balance, capable of weighing to 0.1 mg.

7.0 Procedure

7.1 Preparation of glass fiber filter disc: Place the glass fiber filter on the membrane filter apparatus or insert into bottom of a suitable Gooch crucible with wrinkled surface up. While vacuum is applied, wash the disc with three successive 20 mL volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through. Remove filter from membrane filter apparatus or both crucible and filter if Gooch crucible is used, and dry in an oven at 103-105°C for one hour. Remove to desiccator and store until needed. Repeat the drying cycle until a constant weight is obtained (weight loss is less than 0.5 mg). Weigh immediately before use. After weighing, handle the filter or crucible/filter with forceps or tongs only.

7.2 Selection of Sample Volume
For a 4.7 cm diameter filter, filter 100 mL of sample. If weight of captured residue is less than 1.0 mg, the sample volume must be increased to provide at least 1.0 mg of residue.If other filter diameters are used, start with a sample volume equal to 7 mL/cm^2 of filter area and collect at least a weight of residue proportional to the 1.0 mg stated above.
NOTE: If during filtration of this initial volume the filtration rate drops rapidly, or if filtration time exceeds 5 to 10 minutes, the following scheme is recommended: Use an unweighed glass fiber filter of choice affixed in the filter assembly. Add a known volume of sample to the filter funnel and record the time elapsed after selected volumes have passed through the filter. Twenty-five mL increments for timing are suggested. Continue to record the time and volume increments until filtration rate drops rapidly. Add additional sample if the filter funnel volume is inadequate to reach a reduced rate. Plot the observed time versus volume filtered. Select the proper filtration volume as that just short of the time a significant change in filtration rate occurred.

7.3 Assemble the filtering apparatus and begin suction. Wet the filter with a small volume of distilled water to seat it against the fritted support.

7.4 Shake the sample vigorously and quantitatively transfer the predetermined sample volume selected in 7.2 to the filter using a graduated cylinder. Remove

all traces of water by continuing to apply vacuum after sample has passed through.

7.5 With suction on, wash the graduated cylinder, filter, non-filterable residue and filter funnel wall with three portions of distilled water allowing complete drainage between washing. Remove all traces of water by continuing to apply vacuum after water has passed through.
NOTE: Total volume of wash water used should equal approximately 2 mL per cm^2. For a 4.7 cm filter the total volume is 30 mL.

7.6 Carefully remove the filter from the filter support. Alternatively, remove crucible and filter from crucible adapter. Dry at least one hour at 103-105°C. Cool in a desiccator and weigh. Repeat the drying cycle until a constant weight is obtained (weight loss is less than 0.5 mg).

8.0 Calculations

8.1 Calculate non-filterable residue as follows:

$$\text{Non-filterable residue, mg/L} = \frac{(A - B) \times 1{,}000}{C}$$

where:

A = weight of filter (or filter and crucible) + residue in mg
B = weight of filter (or filter and crucible) in mg
C = mL of sample filtered

9.0 Precision and Accuracy

9.1 Precision data are not available at this time.

9.2 Accuracy data on actual samples cannot be obtained.

Bibliography

1. NCASI Technical Bulletin No. 291, March 1977. National Council of the Paper Industry for Air and Stream Improvement, Inc., 260 Madison Ave., NY.

4. TOTAL PHOSPHORUS

a. Testing Procedures

Testing Methods – Total Phosphorus

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Testing Procedures

Total Phosphorus

TNTplus Low Range 0.05 - 1.50 mg/L PO_4-P, Method 10210 (TNT843)

TNTplus High Range 0.5 - 5.0 mg/L PO_4-P, Method 10210 (TNT844)

TNTplus Ultra High Range 2 - 20 mg/L PO_4-P, Method 10210 (TNT845)

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TNTplus Low Range 0.05 - 1.50 mg/L PO_4-P, Method 10210 (TNT843)

Phosphorus, Reactive (Orthophosphate) and Total

DOC316.53.01124

Ascorbic Acid Method

Method 10209 Reactive
Method 10210 Total

LR (0.15–4.50 mg/L PO_4^{3-} or 0.05–1.50 mg/L PO_4–P)

TNTplus™ 843

Scope and Application: For wastewater, drinking water, boiler water, surface water and process water

Test preparation

How to use instrument-specific information

The *Instrument-specific information* table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

Table 1 Instrument-specific information

Instrument	Light shield
DR 3900	LZV849
DR 3800, DR 2800	LZV646

Before starting the test:

Install the light shield if applicable. See Instrument-specific information.
Please read Safety Advice and Expiration Date on package.
Recommended sample and reagent temperature is 15–25 °C (59–77 °F). Recommended reagent storage temperature is 15–25 °C (59–77 °F).
Recommended sample pH is between 2–10.
The final samples will contain molybdenum. In addition, the final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA. Refer to the current MSDS for safe handling and disposal instructions.
TNT plus methods are activated from the Main Menu screen when the sample vial is inserted into the sample cell holder.

Collect the following items:

Description	Quantity
Phosphorus, Reactive and Total LR TNTplus 843 Reagent Set	1
DRB Reactor for use with 13 mm wells (use adapters with 16 mm Wells)	1
Light Shield (see *Instrument-specific information*)	1
Pipet for 0.2–1.0 mL volumes	1
Pipet Tips for 0.2–1.0 mL pipet	1
Pipet for 1–5 mL volumes	1
Pipet Tips for 1–5 mL pipet	1
Test Tube Rack	1

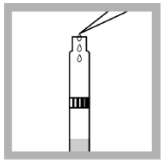
See *Consumables and replacement items* for reorder information.

Total Phosphorus, method 10210

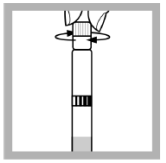
1. Turn on the DRB200 Reactor. Heat to 100 °C.

For DRB200 Reactors with 16-mm wells, insert a16-mm to 13-mm adapter sleeve into each well before turning on the reactor.

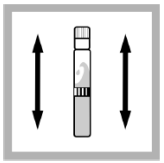
2. Carefully remove the protective foil lid from the DosiCap™ ***Zip***. Unscrew the cap from the vial.



3. Carefully pipet 2.0 mL of sample into the vial.



4. Flip the DosiCap ***Zip*** over so the reagent side faces the vial. Screw the cap tightly onto the vial.



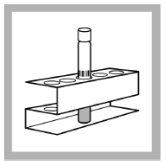
5. Shake the capped vial with 2–3 times to dissolve the reagent in the cap.

Verify that the reagent has dissolved by looking down through the open end of the DosiCap ***Zip***.

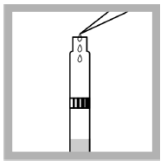
6. Insert the vial in the DRB200 Reactor.

Close the protective cover.

Heat for 1 hour at 100 °C.

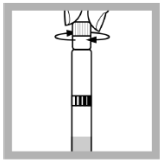


7. After the timer expires, carefully remove the hot vial from the reactor. Insert it in a test tube rack and allow to cool to room temperature (15–25 °C).

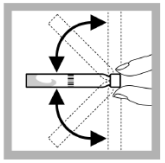


8. Pipet 0.2 mL (200 µL) of Reagent B into the cooled vial.

Immediately close the Reagent B container.

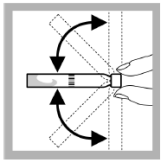


9. Screw a grey DosiCap C onto the vial.



10. Invert the capped vial 2–3 times to dissolve the reagent in the DosiCap.

11. Wait 10 minutes. Install the Light Shield if applicable.



12. When the timer expires, invert the vial again 2–3 times.

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TNTplus Low Range 0.05 - 1.50 mg/L PO_4-P, Method 10210 (TNT843)

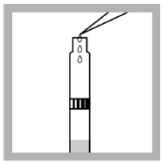
Total Phosphorus, method 10210 (continued)

13. Clean the outside of the vial and insert it into the cell holder. The instrument reads the barcode, then selects and performs the correct test.

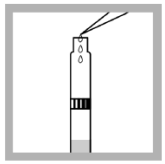
Results are in mg/L PO_4.

No instrument Zero is required.

Reactive Phosphorus, method 10209

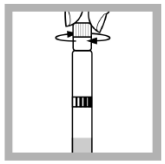


1. Carefully pipet 2.0 mL of sample into the vial.

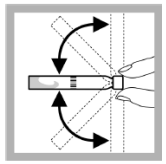


2. Pipet 0.2 mL (200 µL) of Reagent B into the vial.

Immediately close the Reagent B container.



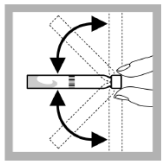
3. Screw a grey DosiCap C onto the vial.



4. Invert the capped vial 2–3 times to dissolve the reagent in the DosiCap.

Reactive Phosphorus, method 10209 (continued)

5. Wait 10 minutes. Install the Light Shield if applicable.



6. When the timer expires, invert the vial again 2–3 times.

7. Clean the outside of the vial and insert it into the cell holder. The instrument reads the barcode, then selects and performs the correct test.

Results are in mg/L PO_4.

No instrument Zero is required.

Reagent blanks

A reagent blank can be measured and the value subtracted from the results of each test performed using the same reagent lot number. Use deionized water in place of sample and perform the *Total Phosphorus, method 10210* or the *Reactive Phosphorus, method 10209* test.

To subtract the value of the blank from a series of measurements:

1. Measure the blank as in step 12 of the *Total Phosphorus, method 10210* test or step 7 of the *Reactive Phosphorus, method 10209* test.
2. Activate the Reagent Blank feature. The measured value of the blank is shown in the highlighted box.
3. Accept the value shown. The reagent blank value will be subtracted from all results until the function is turned off or a different method is selected.

Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

TNTplus Low Range 0.05 - 1.50 mg/L PO_4-P, Method 10210 (TNT843)

Sample blanks

Color or turbidity in samples can cause high results. The digestion in the total phosphate procedure usually destroys all color and turbidity and a sample blank is not required.

To compensate for color or turbidity in the reactive phosphate procedure, the color forming reagent that is present in the DosiCap C is not added.

To determine the sample blank for reactive phosphorus:

1. Run the *Reactive Phosphorus, method 10209* test, but do not add the DosiCap C in step 3.
2. Cap the vial with the original DosiCap ***Zip*** but do not remove the foil. Use the side of the cap without the reagent.
3. Subtract the value obtained in step 7 from the value obtained on the original reactive phosphate sample to give the corrected sample concentration.

Alternatively, reactive phosphate samples that contain only turbidity may be first filtered through a membrane filter and then analyzed. Samples without color or turbidity do not require sample blanks.

Interferences

The ions listed in the *Interfering substances* table have been individually checked up to the given concentrations and do not cause interference. Cumulative effects and the influence of other ions have not been determined. Measurement results can be verified using sample dilutions or standard additions.

Table 2 Interfering substances

Interfering substance	Interference level
SO_4^{2-}	5000 mg/L
Cl^-	2000 mg/L
K^+, Na^+	1000 mg/L
NO_3^-	500 mg/L
Ca^{2+}	250 mg/L
Mg^{2+}	100 mg/L
CO_3^{2-}, Fe^{2+}, Fe^{3+}, Zn^{2+}, Cu^{2+}, Ni^{2+}, I^-, NO_2^-, Cd^{2+}, NH_4^+, Mn^{2+}, Al^{3+}, CO_3^{2-}, SiO_2	50 mg/L
Sn^{4+}, Hg^{2+}	5 mg/L
Ag^+, Pb^{2+}	2.5 mg/L
Cr^{3+}	1 mg/L
Cr^{6+}	0.5 mg/L

Sample collection, preservation and storage

- Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution* and rinsed with deionized water.
- Do not use commercial detergents containing phosphate for cleaning glassware used in this test.
- Analyze samples immediately after collection for best results.
- If prompt analysis is impossible, preserve samples for total phosphorus up to 28 days by adjusting the pH to 2 or less with concentrated Sulfuric Acid* (about 2 mL per liter) and storing at 4 °C.
- Samples to be analyzed for reactive phosphorus should not be preserved with acid: store reactive phosphorus samples at 4 °C and analyze within 48 hours.
- Warm stored samples to 15–25 °C and neutralize with 5.0 N Sodium Hydroxide* before analysis if acid has been added.
- Correct the test results for volume additions.

Accuracy check

Standard solution method
Note: *Refer to the instrument user manual for specific software navigation instructions.*

Required for accuracy check:

- Phosphate standard solution, 3-mg/L
- Wastewater Effluent Mixed Parameters Inorganics Standard

Use one of the following to check accuracy:

- Use 2.0 mL of this 3 mg/L standard in place of the sample in step 3 of the *Total Phosphorus, method 10210* test or step 1 of the *Reactive Phosphorus, method 10209* test.
- Use 2.0 mL of a Wastewater Effluent Mixed Parameters Inorganics Standard in place of the sample in step 3 of the *Total Phosphorus, method 10210* test or step 1 of the *Reactive Phosphorus, method 10209* test. This standard contains 2 mg/L phosphate in the presence of several other ions such as nitrate, sulfate and ammonia.

Method performance

Program	Standard	Precision 95% Confidence Limits of Distribution
Barcode	3.50 mg/L PO_4	3.39–3.61 mg/L PO_4

* See *Optional reagents and apparatus.*

TNTplus Low Range 0.05 - 1.50 mg/L PO_4-P, Method 10210 (TNT843)

Summary of method

Phosphates present in organic and condensed inorganic forms (meta-, pyro- or other polyphosphates) are first converted to reactive orthophosphate in the total phosphorus procedure. Treatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are also converted to orthophosphates in the total phosphorus procedure by heating with acid and persulfate. The reactive phosphorus procedure measures only the reactive (ortho) phosphorus present in the sample.

The reactive or orthophosphate ions react with molybdate and antimony ions in an acidic solution to form an antimonyl phosphomolybdate complex, which is reduced by ascorbic acid to phosphomolybdenum blue. Test results are measured at 890 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
Phosphorus, Reactive and Total, LR TNT843 Reagent Set	1	25/pkg	TNT843

Required apparatus

Description	Quantity	Unit	Catalog number
DRB200 Reactor, 115 V, 9x13mm + 2x20 mm (mono block)	1	each	DRB20001
OR			
DRB200 Reactor, 230 V, 9x13mm + 2x20 mm (mono block)	1	each	DRB20005
Light shield, DR 3900	1	each	LZV849
Light shield, DR 3800, DR 2800	1	each	LZV646
Pipet, variable volume, 0.2–1.0 mL	1	each	BBP078
Pipet Tips, for BBP078 pipet	1	100/pkg	BBP079
Pipet, variable volume, 1–5 mL	1	each	BBP065
Pipet Tips, for BBP065 pipet	1	75/pkg	BBP068
Test Tube Rack	1	each	1864100

Recommended standards and apparatus

Description	Unit	Catalog number
Phosphate Standard Solution, 3-mg/L as PO_4^{3-}	946 mL	2059716
Wastewater Effluent Inorganics Standard for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	2833249

Optional reagents and apparatus

Description	Unit	Catalog number
TNTplus Reactor adapter sleeves, 16-mm to 13-mm diameter	5/pkg	2895805
Bottle, sampling, low density poly, w/cap, 500 mL	12/pkg	2087079
DRB200 Reactor, 115 V, 21x13 mm + 4x20 mm (dual block)	each	DRB20002
DRB200 Reactor, 115 V, 15x13 mm + 15x13 mm (dual block)	each	DRB20003
DRB200 Reactor, 115 V, 12x13 mm + 8x20 mm (dual block)	each	DRB20004
DRB200 Reactor, 230 V, 21x13 mm + 4x20 mm (dual block)	each	DRB20006
DRB200 Reactor, 230 V, 15x13 mm + 15x13 mm (dual block)	each	DRB20007
DRB200 Reactor, 230 V, 12x13 mm + 8x20 mm (dual block)	each	DRB20008
Filter Holder, glass for vacuum filtration	each	234000
Filter, membrane, 47-mm, 0.45-micron, hydrophilic, polyethersulfone	each	2894700
Flask, filtering, glass, 1000-mL	each	54653
Hydrochloric Acid 6N (1:1)	500 mL	88449
Sodium Hydroxide, 5.0 N	1000 mL	245053
Sulfuric Acid, concentrated	500 mL	97949
Tubing, rubber	12 ft	56019
pH Paper, 0–14 pH range	100/pkg	2601300
Thermometer, Non-Mercury, -10 to 225 °C	each	2635700
Finger cots	2/pkg	1464702
Pipet, serological, 2 mL	each	53236

Optional standards

Description	Unit	Catalog number
Voluette Ampule breaker 10 mL	each	2196800
Phosphate, 15 mg/L	100 mL	1424342
Phosphate, 30 mg/L	946 mL	1436716
Phosphate, 50 mg/L, 10 mL Voluette Ampules	16/pkg	17110
Phosphate, 100 mg/L	100 mL	1436832
Phosphate, 500 mg/L, 10 mL Voluette Ampules	16/pkg	1424210
Phosphate, 500 mg/L	100 mL	1424232

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Edition 6

TNTplus Low Range 0.05 - 1.50 mg/L PO_4-P, Method 10210 (TNT843)

Remove foil cap and unscrew DosiCap Zip

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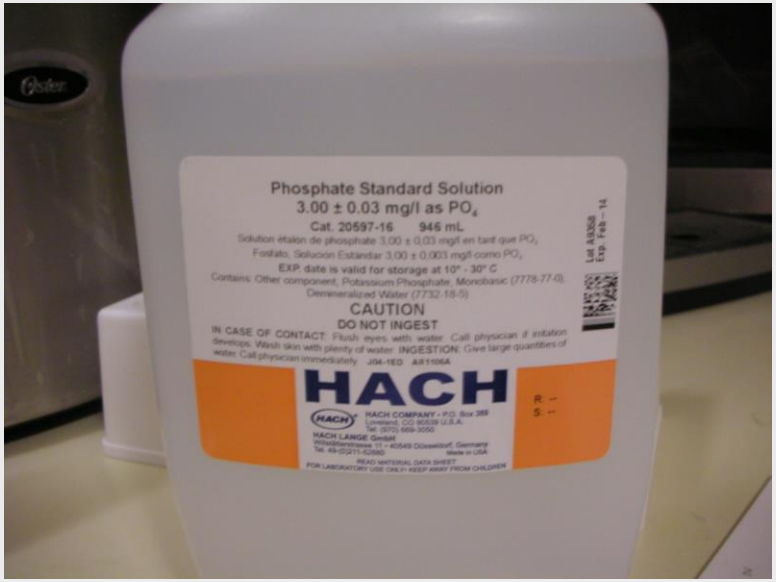

Quality Assurance Phosphate Standard Solution 3.00 ± 0.03 mg/L as PO_4

Add 2.0 mL of Phosphate Standard to vial

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TNTplus Low Range 0.05 - 1.50 mg/L PO_4-P, Method 10210 (TNT843)

Add 2.0 mL of sample to vial

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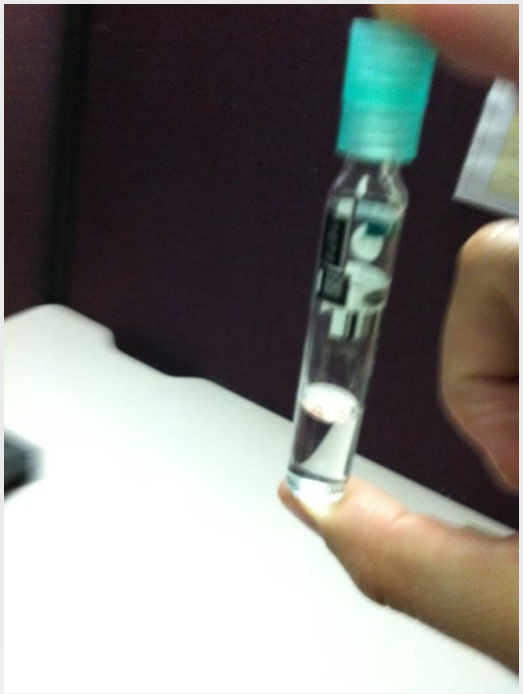
TNTplus Low Range 0.05 - 1.50 mg/L PO_4-P, Method 10210 (TNT843)

Flip over the DosiCap Zip and tighten



Shake the capped vial 2 to 3 times in an up and downward motion

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TNTplus Low Range 0.05 - 1.50 mg/L PO_4-P, Method 10210 (TNT843)

Insert vial or vials into the DRB200 Reactor
Close cover and heat for 1 hour at 60° C

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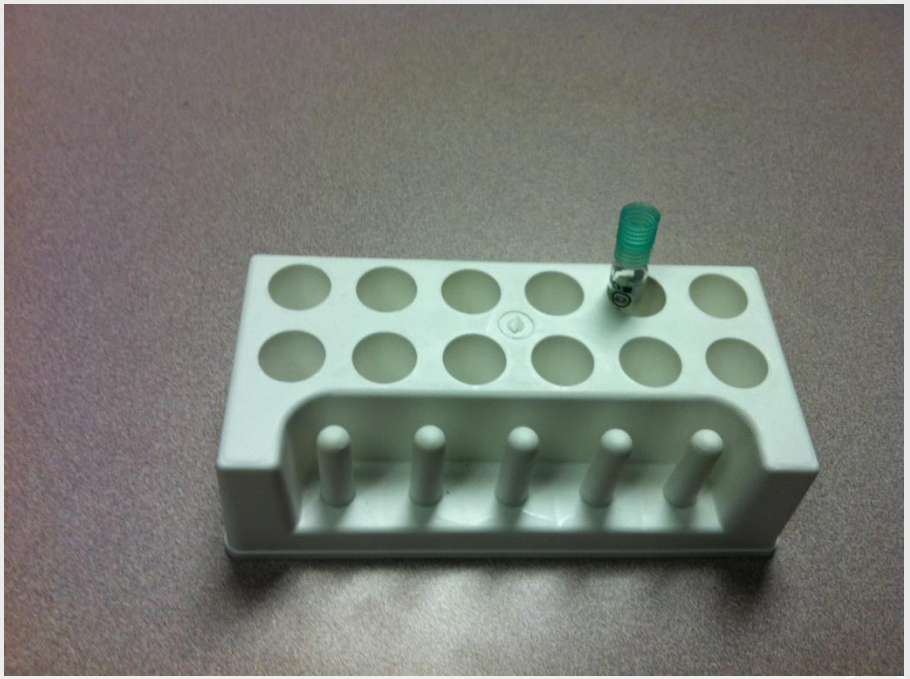
After the timer expires carefully remove the hot vial or vials form the reactor. Insert vial or vials in a test tube rack and allow to cool to room temperature.



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Pipet 0.2 mL of Reagent B into the cooled vial

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TNTplus Low Range 0.05 - 1.50 mg/L PO_4-P, Method 10210 (TNT843)

Screw a grey DosiCap C onto the vial

Invert the capped vial 2-3 times to dissolve the reagent in the DosiCap
Wait 10 minutes.

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TNTplus Low Range 0.05 - 1.50 mg/L PO_4-P, Method 10210 (TNT843)

When the timer expires invert again 2-3 times and then insert into a Spectrophotometer for readings

Record the readings

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TNTplus High Range 0.5 - 5.0 mg/L PO_4-P, Method 10210 (TNT844)

Phosphorus, Reactive (Orthophosphate) and Total

DOC316.53.01125

Ascorbic Acid Method

Method 10209 Reactive
Method 10210 Total

HR (1.5 to 15.0 mg/L PO_4^{3-} or 0.5 to 5.0 mg/L PO_4–P)

TNTplus™ 844

Scope and Application: For wastewater, drinking water, boiler water, surface water and process analysis

Test preparation

How to use instrument-specific information

The *Instrument-specific information* table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

Table 1 Instrument-specific information

Instrument	Light shield
DR 3900	LZV849
DR 3800, DR 2800	LZV646

Before starting the test:

DR 3900, DR 3800, DR 2800: Install the light shield in Cell Compartment #2 before performing this test.
Please read Safety Advice and Expiration Date on package.
Recommended sample and reagent temperature is 15–25 °C (59–77 °F). Recommended reagent storage temperature is 15–25 °C (59–77 °F).
Recommended sample pH is between 2–10.
The final samples will contain molybdenum. In addition, the final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA. Refer to the current MSDS for safe handling and disposal instructions.
TNT plus methods are activated from the Main Menu screen when the sample vial is inserted into the sample cell holder.

Collect the following items:

Description	Quantity
Phosphorus, Reactive Total HR TNT 844 Reagent Set	1
DRB Reactor for use with 13 mm wells (use adapters with 16 mm wells)	1
Light Shield (see *Instrument-specific information*)	1
Pipet for 0.2–1.0 mL volumes	1
Pipet Tips for 0.2–1.0 mL pipet	2
Test Tube Rack	1

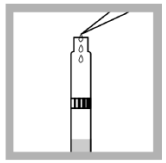
See *Consumables and replacement items* for reorder information.

Total Phosphorus, method 10210

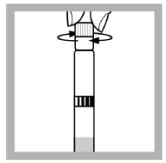
1. Turn on the DRB200 Reactor. Heat to 100 °C.

For DRB200 Reactors with 16-mm wells, insert a16-mm to 13-mm adapter sleeve into each well before turning on the reactor.

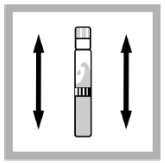
2. Carefully remove the protective foil lid from the DosiCap™ ***Zip***. Unscrew the cap from the vial.



3. Carefully pipet 0.5 mL (500 µL) of sample into the vial.



4. Flip the DosiCap ***Zip*** over so the reagent side faces the vial. Screw the cap tightly onto the vial.



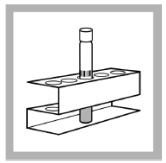
5. Shake the capped vial 2–3 times to dissolve the reagent in the cap.

Verify that the reagent has dissolved by looking down through the open end of the DosiCap ***Zip***.

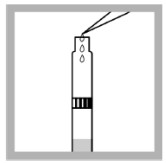
6. Insert the vial in the DRB200 Reactor.

Close the protective cover.

Heat for 1 hour at 100 °C.

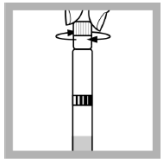


7. After the timer expires, carefully remove the hot vial from the reactor. Insert it in a test tube rack and allow to cool to room temperature (15–25 °C).

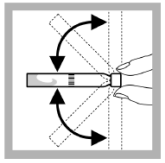


8. Pipet 0.2 mL (200 µL) of Reagent B into the cooled vial.

Immediately close the Reagent B container.

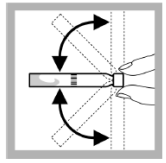


9. Screw a grey DosiCap C onto the vial.



10. Invert the capped vial 2–3 times to dissolve the reagent in the DosiCap.

11. Wait 10 minutes. Install the Light Shield if applicable.



12. When the timer expires, invert the vial again 2–3 times.

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TNTplus High Range 0.5 - 5.0 mg/L PO_4-P, Method 10210 (TNT844)

Total Phosphorus, method 10210 (continued)

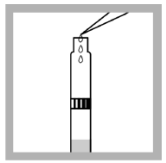
13. Clean the outside of the vial and insert it into the cell holder. The instrument reads the barcode, then selects and performs the correct test.

Results are in mg/L PO_4.

No instrument Zero is required.

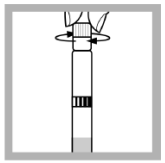
Reactive Phosphorus, method 10209

1. Carefully pipet 0.5 mL of sample into the vial.

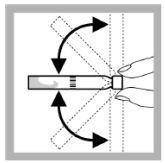


2. Pipet 0.2 mL (200 µL) of Reagent B into the vial.

Immediately close the Reagent B container.



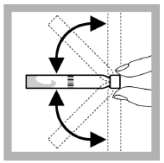
3. Screw a grey DosiCap C onto the vial.



4. Invert the capped vial 2–3 times to dissolve the reagent in the DosiCap.

Reactive Phosphorus, method 10209 (continued)

5. Wait 10 minutes. Install the Light Shield if applicable.



6. When the timer expires, invert the vial again 2–3 times.

7. Clean the outside of the vial and insert it into the cell holder. The instrument reads the barcode, then selects and performs the correct test.

Results are in mg/L PO_4.

No instrument Zero is required.

Reagent blanks

A reagent blank can be measured and the value subtracted from the results of each test performed using the same reagent lot number. Use deionized water in place of sample and perform the *Total Phosphorus, method 10210* or the *Reactive Phosphorus, method 10209* test.

To subtract the value of the blank from a series of measurements:

1. Measure the blank as in step 13 of the *Total Phosphorus, method 10210* test or step 7 of the *Reactive Phosphorus, method 10209* test.
2. Activate the Reagent Blank feature. The measured value of the blank is shown in the highlighted box.
3. Accept the value shown. The reagent blank value will be subtracted from all results until the function is turned off or a different method is selected.

Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

Sample blanks

Color or turbidity in samples can cause high results. The digestion in the total phosphate procedure usually destroys all color and turbidity and a sample blank is not required.

To compensate for color or turbidity in the reactive phosphate procedure, the color forming reagent that is present in the DosiCap C is not added.

To determine the sample blank for reactive phosphorus:

1. Run the *Reactive Phosphorus, method 10209* test, but do not add the DosiCap C in step 3.
2. Cap the vial with the original DosiCap ***Zip*** but do not remove the foil. Use the side of the cap without the reagent.
3. Subtract the value obtained in step 7 from the value obtained on the original reactive phosphate sample to give the corrected sample concentration.

TNTplus High Range 0.5 - 5.0 mg/L PO_4-P, Method 10210 (TNT844)

Alternatively, reactive phosphate samples that contain only turbidity may be first filtered through a membrane filter and then analyzed. Samples without color or turbidity do not require sample blanks.

Interferences

The ions listed in the *Interfering substances* table have been individually checked up to the given concentrations and do not cause interference. Cumulative effects and the influence of other ions have not been determined. Measurement results can be verified using sample dilutions or standard additions.

Table 2 Interfering substances

Interfering substance	Interference level
SO_4^{2-}	20 g/L
Cl^-	10 g/L
Ca^{2+}	1000 mg/L
K^+, Na^+	4000 mg/L
NO_3^-	500 mg/L
Mg^{2+}	400 mg/L
Co^{2+}, Fe^{2+}, Fe^{3+}, Zn^{2+},Cu^{2+}, Ni^{2+}, NO_2^-, Cd^{2+}, NH^{4+}, Mn^{2+}, Al^{3+}, CO_3^{2-}	200 mg/L
I^-	100 mg/L
SiO_2	50 mg/L
Hg^{2+}	40 mg/L
Pb^{2+}	20 mg/L
Ag^+, Sn^{4+}	10 mg/L
Cr^{3+}	5 mg/L
Cr^{6+}	1 mg/L

Sample collection, preservation and storage

- Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution* and rinsed with deionized water.
- Do not use commercial detergents containing phosphate for cleaning glassware used in this test.
- Analyze samples immediately after collection for best results.
- If prompt analysis is impossible, preserve samples for total phosphorus up to 28 days by adjusting the pH to 2 or less with concentrated Sulfuric Acid* (about 2 mL per liter) and storing at 4 °C.
- Samples to be analyzed for reactive phosphorus should not be preserved with acid: store reactive phosphorus samples at 4 °C and analyze within 48 hours.
- Warm stored samples to 15–25 °C and neutralize with 5.0 N Sodium Hydroxide* before analysis if acid has been added.
- Correct the test results for volume additions.

* See *Optional reagents and apparatus*.

Accuracy check

Standard solution method
Note: *Refer to the instrument user manual for specific software navigation instructions.*

Required for accuracy check:

- Phosphate standard solution, 10-mg/L
- Wastewater Influent Mixed Parameters Inorganics Standard

Use one of the following to check accuracy:

- Use 0.5 mL of this 10 mg/L standard in place of the sample in step 3 of the *Total Phosphorus, method 10210* test or step 1 of the *Reactive Phosphorus, method 10209* test.
- Use 0.5 mL of a Wastewater Influent Mixed Parameters Inorganics Standard in place of the sample in step 3 of the *Total Phosphorus, method 10210* test or step 1 of the *Reactive Phosphorus, method 10209* test. This standard contains 10 mg/L phosphate in the presence of several other ions such as nitrate, sulfate and ammonia.

Method performance

Program	Standard	Precision 95% Confidence Limits of Distribution
Barcode	10.0 mg/L PO_4	9.5–10.5 mg/L PO_4

Summary of method

Phosphates present in organic and condensed inorganic forms (meta-, pyro- or other polyphosphates) are first converted to reactive orthophosphate in the total phosphorus procedure. Treatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are also converted to orthophosphates in the total phosphorus procedure by heating with acid and persulfate. The reactive phosphorus procedure measures only the reactive (ortho) phosphorus present in the sample.

The reactive or orthophosphate ions react with molybdate and antimony ions in an acidic solution to form an antimonyl phosphomolybdate complex, which is reduced by ascorbic acid to phosphomolybdenum blue. Test results are measured at 890 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
Phosphorus, Reactive and Total, HR TNT844 Reagent Set	1	25/pkg	TNT844

Required apparatus

Description	Quantity	Unit	Catalog number
DRB200 Reactor, 115 V, 9x13mm + 2x20 mm (mono block)	1	each	DRB20001
OR			
DRB200 Reactor, 230 V, 9x13mm + 2x20 mm (mono block)	1	each	DRB20005
Light Shield, DR 3800, DR 2800	1	each	LZV646
Light Shield, DR 3900	1	each	LZV849

TNTplus High Range 0.5 - 5.0 mg/L PO_4-P, Method 10210 (TNT844)

Required apparatus (continued)

Description	Quantity	Unit	Catalog number
Pipet, variable volume, 0.2–1.0 mL	1	each	BBP078
Pipet Tips, for BBP078 pipet	1	100/pkg	BBP079
Test Tube Rack	1	each	1864100

Recommended standards and apparatus

Description	Unit	Catalog number
Phosphate Standard Solution, 10-mg/L as PO_4	946 mL	1420416
Wastewater Influent Inorganics Standard for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	2833149

Optional reagents and apparatus

Description	Unit	Catalog number
TNTplus Reactor adapter sleeves, 16-mm to 13-mm diameter	5/pkg	2895805
Bottle, sampling, low density poly, w/cap, 500 mL	12/pkg	2087079
DRB200 Reactor, 115 V, 21x13 mm + 4x20 mm (dual block)	each	DRB20002
DRB200 Reactor, 115 V, 15x13 mm + 15x13 mm (dual block)	each	DRB20003
DRB200 Reactor, 115 V, 12x13 mm + 8x20 mm (dual block)	each	DRB20004
DRB200 Reactor, 230 V, 21x13 mm + 4x20 mm (dual block)	each	DRB20006
DRB200 Reactor, 230 V, 15x13 mm + 15x13 mm (dual block)	each	DRB20007
DRB200 Reactor, 230 V, 12x13 mm + 8x20 mm (dual block)	each	DRB20008
Filter Holder, glass for vacuum filtration	each	234000
Filter, membrane, 47-mm, 0.45-micron, hydrophilic, polyethersulfone	each	2894700
Flask, filtering, glass, 1000-mL	each	54653
Hydrochloric Acid 6N (1:1)	500 mL	88449
Sodium Hydroxide, 5.0 N	1000 mL	245053
Sulfuric Acid, concentrated	500 mL	97949
Tubing, rubber	12 ft	56019
pH Paper, 0–14 pH range	100/pkg	2601300
Thermometer, Non-Mercury, -10 to 225 °C	each	2635700
Finger cots	2/pkg	1464702
Pipet, serological, 2 mL	each	53236

Optional standards

Description	Unit	Catalog number
Voluette Ampule breaker 10 mL	each	2196800
Phosphate, Standard Solution, 15 mg/L	100 mL	1424342
Phosphate, Standard Solution, 30 mg/L	946 mL	1436716
Phosphate, Standard Solution, 50 mg/L, 10 mL Voluette Ampules	16/pkg	17110
Phosphate, Standard Solution, 100 mg/L	100 mL	1436832
Phosphate, Standard Solution, 500 mg/L, 10 mL Voluette Ampules	16/pkg	1424210
Phosphate, Standard Solution, 500 mg/L	100 mL	1424232

TNTplus High Range 0.5 - 5.0 mg/L PO_4-P, Method 10210 (TNT844)

Remove foil cap and unscrew DosiCap Zip

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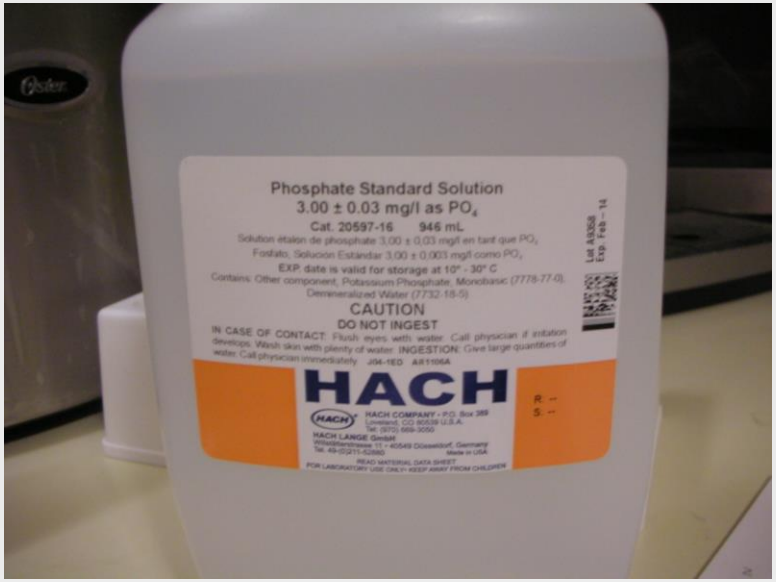

Quality Assurance Phosphate Standard Solution 3.00 ± 0.03 mg/L as PO_4

Add 0.5 mL of Phosphate Standard to vial

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Add 0.5 mL of sample to vial

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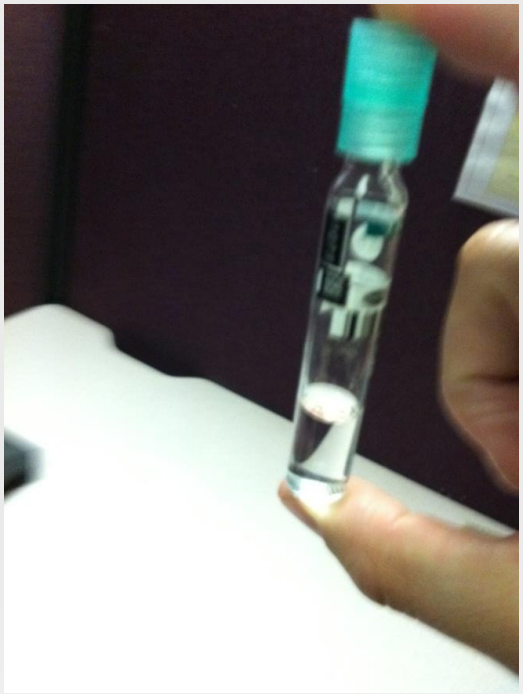
TNTplus High Range 0.5 - 5.0 mg/L PO_4-P, Method 10210 (TNT844)

Flip over the DosiCap Zip and tighten



Shake the capped vial 2 to 3 times in an up and downward motion

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Insert vial or vials into the DRB200 Reactor
Close cover and heat for 1 hour at 60° C

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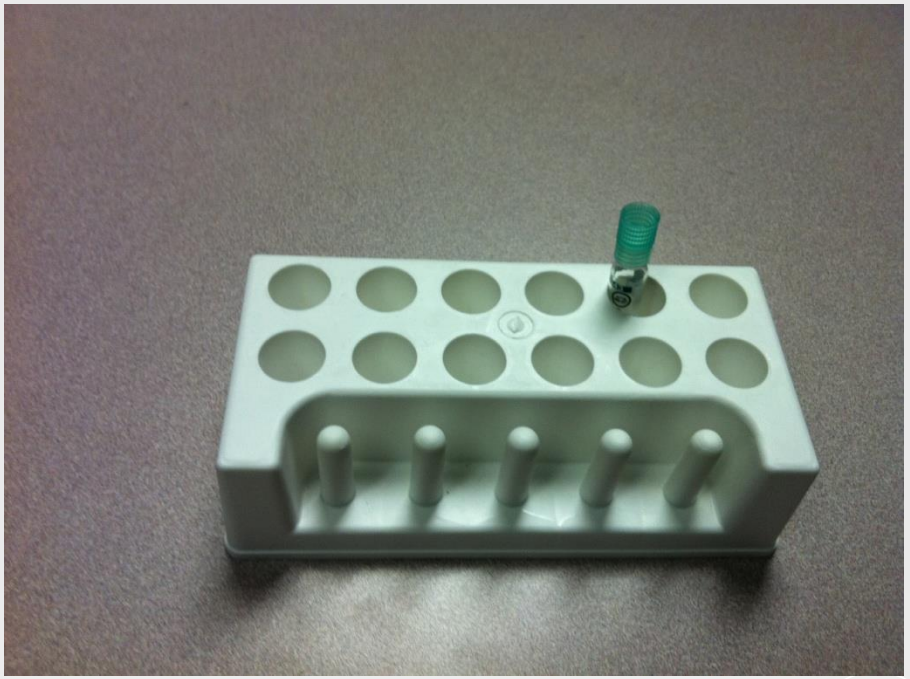
After the timer expires carefully remove the hot vial or vials form the reactor. Insert vial or vials in a test tube rack and allow to cool to room temperature.



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TNTplus High Range 0.5 - 5.0 mg/L PO_4-P, Method 10210 (TNT844)

Pipet 0.2 mL of Reagent B into the cooled vial

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TNTplus High Range 0.5 - 5.0 mg/L PO_4-P, Method 10210 (TNT844)

Screw a grey DosiCap C onto the vial

Invert the capped vial 2-3 times to dissolve the reagent in the DosiCap
Wait 10 minutes.

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When the timer expires invert again 2-3 times and then insert into a Spectrophotometer for readings

Record the readings

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TNTplus Ultra High Range 2 - 20 mg/L PO_4-P, Method 10210 (TNT845)

Phosphorus, Reactive (Orthophosphate) and Total

DOC316.53.01126

Ascorbic Acid Method

Method 10209 Reactive
Method 10210 Total

UHR (6 to 60 mg/L PO_4^{3-} or 2 to 20 mg/L PO_4–P)

TNTplus™ 845

Scope and Application: For wastewater, drinking water, boiler water, surface water and process analysis

Test preparation

How to use instrument-specific information

The *Instrument-specific information* table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

Table 1 Instrument-specific information

Instrument	Light shield
DR 3900	LZV849
DR 3800, DR 2800	LZV646

Before starting the test:

DR 3900, DR 3800, DR 2800: Install the light shield in Cell Compartment #2 before performing this test.
Please read Safety Advice and Expiration Date on package.
Recommended sample and reagent temperature is 15–25 °C (59–77 °F). Recommended reagent storage temperature is 15–25 °C (59–77 °F).
Recommended sample pH is between 2–10.
The final samples will contain molybdenum. In addition, the final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA. Refer to the current MSDS for safe handling and disposal instructions.
TNT plus methods are activated from the Main Menu screen when the sample vial is inserted into the sample cell holder.

Collect the following items:

Description	Quantity
Phosphorus, Reactive Total HR TNT845 Reagent Set	1
DRB Reactor for use with 13 mm wells (use adapters with 16 mm wells)	1
Light Shield (see *Instrument-specific information*)	1
Pipet for 0.2–1.0 mL volumes	1
Pipet Tips for 0.2–1.0 mL pipet	2
Test Tube Rack	1

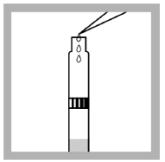
See *Consumables and replacement items* for reorder information.

Total Phosphorus, method 10210

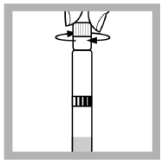
1. Turn on the DRB200 Reactor. Heat to 100 °C.

For DRB200 Reactors with 16-mm wells, insert a16-mm to 13-mm adapter sleeve into each well before turning on the reactor.

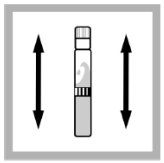
2. Carefully remove the protective foil lid from the DosiCap™ ***Zip***. Unscrew the cap from the vial.



3. Carefully pipet 0.4 mL (400 µL) of sample into the vial.



4. Flip the DosiCap ***Zip*** over so the reagent side faces the vial. Screw the cap tightly onto the vial.



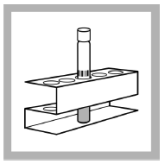
5. Shake the capped vial 2–3 times to dissolve the reagent in the cap.

Verify that the reagent has dissolved by looking down through the open end of the DosiCap ***Zip***.

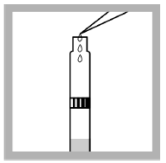
6. Insert the vial in the DRB200 Reactor.

Close the protective cover.

Heat for 1 hour at 100 °C.

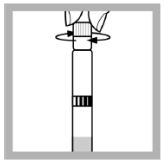


7. After the timer expires, carefully remove the hot vial from the reactor. Insert it in a test tube rack and allow to cool to room temperature (15–25 °C).

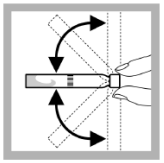


8. Pipet 0.5 mL (500 µL) of Reagent B into the cooled vial.

Immediately close the Reagent B container.

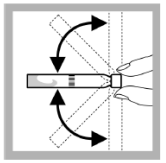


9. Screw a grey DosiCap C onto the vial.



10. Invert the capped vial 2–3 times to dissolve the reagent in the DosiCap.

11. Wait 10 minutes. Install Light Shield if applicable.



12. When the timer expires, invert the vial again 2–3 times.

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TNTplus Ultra High Range 2 - 20 mg/L PO_4-P, Method 10210 (TNT845)

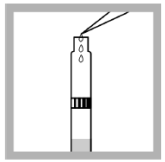
Total Phosphorus, method 10210 (continued)

13. Clean the outside of the vial and insert it into the cell holder. The instrument reads the barcode, then selects and performs the correct test.

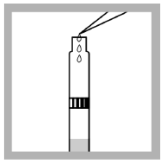
Results are in mg/L PO_4.

No instrument Zero is required.

Reactive Phosphorus, method 10209

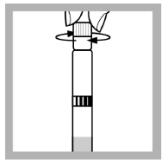


1. Carefully pipet 0.4 mL of sample into the vial.

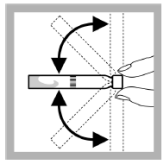


2. Pipet 0.5 mL (500 µL) of Reagent B into the vial.

Immediately close the Reagent B container.



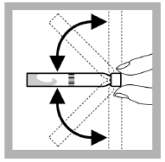
3. Screw a grey DosiCap C onto the vial.



4. Invert the capped vial 2–3 times to dissolve the reagent in the DosiCap.

Reactive Phosphorus, method 10209 (continued)

5. Wait 10 minutes. Install the Light Shield if applicable.



6. When the timer expires, invert the vial again 2–3 times.

7. Clean the outside of the vial and insert it into the cell holder. The instrument reads the barcode, then selects and performs the correct test.

Results are in mg/L PO_4.

No instrument Zero is required.

Reagent blanks

A reagent blank can be measured and the value subtracted from the results of each test performed using the same reagent lot number. Use deionized water in place of sample and perform the *Total Phosphorus, method 10210* or the *Reactive Phosphorus, method 10209* test.

To subtract the value of the blank from a series of measurements:

1. Measure the blank as in step 13 of the *Total Phosphorus, method 10210* test or step 7 of the *Reactive Phosphorus, method 10209* test.
2. Activate the Reagent Blank feature. The measured value of the blank is shown in the highlighted box.
3. Accept the value shown. The reagent blank value will be subtracted from all results until the function is turned off or a different method is selected.

Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

Sample blanks

Color or turbidity in samples can cause high results. The digestion in the total phosphate procedure usually destroys all color and turbidity and a sample blank is not required.

To compensate for color or turbidity in the reactive phosphate procedure, the color forming reagent that is present in the DosiCap C is not added.

To determine the sample blank for reactive phosphorus:

1. Run the *Reactive Phosphorus, method 10209* test, but do not add the DosiCap C in step 3.
2. Cap the vial with the original DosiCap ***Zip*** but do not remove the foil. Use the side of the cap without the reagent.
3. Subtract the value obtained in step 7 from the value obtained on the original reactive phosphate sample to give the corrected sample concentration.

TNTplus Ultra High Range 2 - 20 mg/L PO_4-P, Method 10210 (TNT845)

Alternatively, reactive phosphate samples that contain only turbidity may be first filtered through a membrane filter and then analyzed. Samples without color or turbidity do not require sample blanks.

Interferences

The ions listed in the *Interfering substances* table have been individually checked up to the given concentrations and do not cause interference. Cumulative effects and the influence of other ions have not been determined. Measurement results can be verified using sample dilutions or standard additions.

Table 2 Interfering substances

Interfering substance	Interference level
SO_4^{2-}	5000 mg/L
Cl^-	2000 mg/L
K^+, Na^+, Ca^{2+}	1000 mg/L
Mg^{2+}, NO_3^-	500 mg/L
Co^{2+}, Fe^{2+}, Fe^{3+}, Zn^{2+},Cu^{2+}, Ni^{2+}, I^-, NO_2^-, Cd^{2+}, Sn^{4+},NH_4^+, Mn^{2+}, Al^{3+}, Hg^{2+}, Pb^{2+}, SiO_2	50 mg/L
Ag^+	25 mg/L
Cr^{3+}	10 mg/L
Cr^{6+}	5 mg/L

Sample collection, preservation and storage

- Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution* and rinsed with deionized water.
- Do not use commercial detergents containing phosphate for cleaning glassware used in this test.
- Analyze samples immediately after collection for best results.
- If prompt analysis is impossible, preserve samples for total phosphorus up to 28 days by adjusting the pH to 2 or less with concentrated Sulfuric Acid* (about 2 mL per liter) and storing at 4 °C.
- Samples to be analyzed for reactive phosphorus should not be preserved with acid: store reactive phosphorus samples at 4 °C and analyze within 48 hours.
- Warm stored samples to 15–25 °C and neutralize with 5.0 N Sodium Hydroxide* before analysis if acid has been added.
- Correct the test results for volume additions.

Accuracy check

Standard solution method

Note: *Refer to the instrument user manual for specific software navigation instructions.*

Required for accuracy check:

- Phosphate standard solution, 50-mg/L
- Wastewater Influent Mixed Parameters Inorganics Standard

* See *Optional reagents and apparatus*.

Use one of the following to check accuracy:

- Use 0.4 mL of this 50 mg/L standard in place of the sample in step 3 of the *Total Phosphorus, method 10210* test or step 1 of the *Reactive Phosphorus, method 10209* test.
- Use 0.4 mL of a Wastewater Influent Mixed Parameters Inorganics Standard in place of the sample in step 3 of the *Total Phosphorus, method 10210* test or step 1 of the *Reactive Phosphorus, method 10209* test. This standard contains 10 mg/L phosphate in the presence of several other ions such as nitrate, sulfate and ammonia.

Method performance

Program	Standard	Precision 95% Confidence Limits of Distribution
90	50.0 mg/L PO_4	49–51 mg/L PO_4

Summary of method

Phosphates present in organic and condensed inorganic forms (meta-, pyro- or other polyphosphates) are first converted to reactive orthophosphate in the total phosphorus procedure. Treatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are also converted to orthophosphates in the total phosphorus procedure by heating with acid and persulfate. The reactive phosphorus procedure measures only the reactive (ortho) phosphorus present in the sample.

The reactive or orthophosphate ions react with molybdate and antimony ions in an acidic solution to form an antimonyl phosphomolybdate complex, which is reduced by ascorbic acid to phosphomolybdenum blue. Test results are measured at 890 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
Phosphorus, Reactive and Total, UHR TNT845 Reagent Set	1	25/pkg	TNT845

Required apparatus

Description	Quantity	Unit	Catalog number
DRB200 Reactor, 115 V, 9x13mm + 2x20 mm (mono block)	1	each	DRB20001
OR			
DRB200 Reactor, 230 V, 9x13mm + 2x20 mm (mono block)	1	each	DRB20005
Light shield, DR 3800, DR 2800	1	each	LZV646
Light shield, DR 2900	1	each	LZV849
Pipet, variable volume, 0.2–1.0 mL	1	each	BBP078
Pipet Tips, for BBP078 pipet	2	100/pkg	BBP079
Test Tube Rack	1–3	each	1864100

TNTplus Ultra High Range 2 - 20 mg/L PO_4-P, Method 10210 (TNT845)

Recommended standards and apparatus

Description	Unit	Catalog number
Phosphate Standard Solution, 50-mg/L as PO_4	500 mL	17149
Wastewater Influent Inorganics Standard for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	2833149

Optional reagents and apparatus

Description	Unit	Catalog number
TNTplus Reactor adapter sleeves, 16-mm to 13-mm diameter	5/pkg	2895805
Bottle, sampling, low density poly, w/cap, 500 mL	12/pkg	2087079
DRB200 Reactor, 115 V, 21x13 mm + 4x20 mm (dual block)	each	DRB20002
DRB200 Reactor, 115 V, 15x13 mm + 15x13 mm (dual block)	each	DRB20003
DRB200 Reactor, 115 V, 12x13 mm + 8x20 mm (dual block)	each	DRB20004
DRB200 Reactor, 230 V, 21x13mm + 4x20 mm (dual block)	each	DRB20006
DRB200 Reactor, 230 V, 15x13mm + 15x13 mm (dual block)	each	DRB20007
DRB200 Reactor, 230 V, 12x13mm + 8x20 mm (dual block)	each	DRB20008
Filter Holder, glass for vacuum filtration	each	234000
Filter, membrane, 47-mm, 0.45-micron, hydrophilic, polyethersulfone	each	2894700
Flask, filtering, glass, 1000-mL	each	54653
Hydrochloric Acid 6N (1:1)	500 mL	88449
Sodium Hydroxide, 5.0 N	1000 mL	245053
Sulfuric Acid, concentrated	500 mL	97949
Tubing, rubber	12 ft	56019
pH Paper, 0–14 pH range	100/pkg	2601300
Thermometer, Non-Mercury, -10 to 225 °C	each	2635700
Finger cots	2/pkg	1464702
Pipet, serological, 2 mL	each	53236

Optional standards

Description	Unit	Catalog number
Voluette Ampule breaker 10 mL	each	2196800
Phosphate, Standard Solution, 15 mg/L	100 mL	1424342
Phosphate, Standard Solution, 30 mg/L	946 mL	1436716
Phosphate, Standard Solution, 50 mg/L, 10 mL Voluette Ampules	16/pkg	17110
Phosphate, Standard Solution, 100 mg/L	100 mL	1436832
Phosphate, Standard Solution, 500 mg/L, 10 mL Voluette Ampules	16/pkg	1424210
Phosphate, Standard Solution, 500 mg/L	100 mL	1424232

TNTplus Ultra High Range 2 - 20 mg/L PO_4-P, Method 10210 (TNT845)

Remove foil cap and unscrew DosiCap Zip

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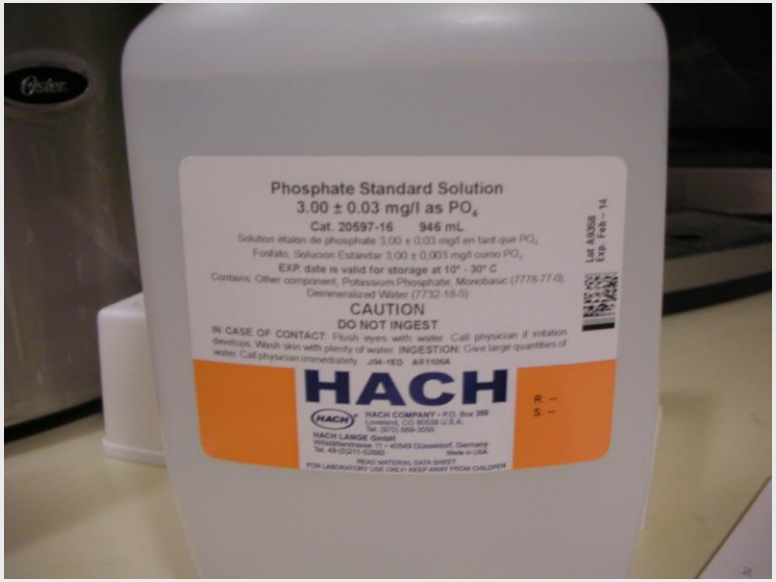
TNTplus Ultra High Range 2 - 20 mg/L PO_4-P, Method 10210 (TNT845)



Quality Assurance Phosphate Standard Solution 3.00 ± 0.03 mg/L as PO_4

Add 0.4 mL of Phosphate Standard to vial

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TNTplus Ultra High Range 2 - 20 mg/L PO_4-P, Method 10210 (TNT845)

Add 0.4 mL of sample to vial

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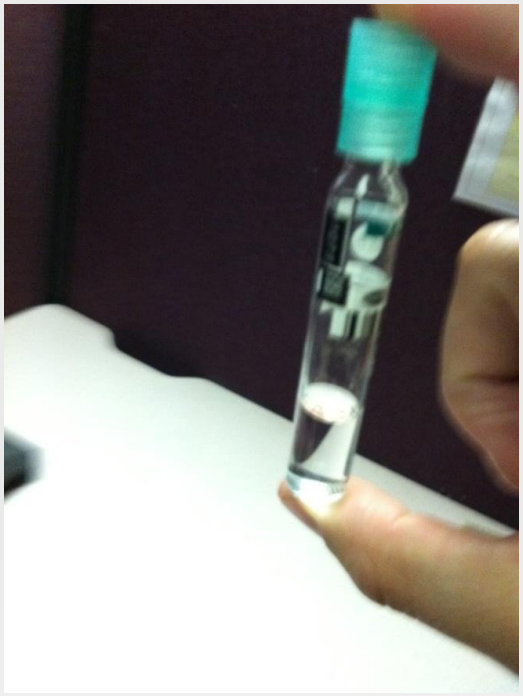
TNTplus Ultra High Range 2 - 20 mg/L PO_4-P, Method 10210 (TNT845)

Flip over the DosiCap Zip and tighten



Shake the capped vial 2 to 3 times in an up and downward motion

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TNTplus Ultra High Range 2 - 20 mg/L PO_4-P, Method 10210 (TNT845)

Insert vial or vials into the DRB200 Reactor
Close cover and heat for 1 hour at 60° C

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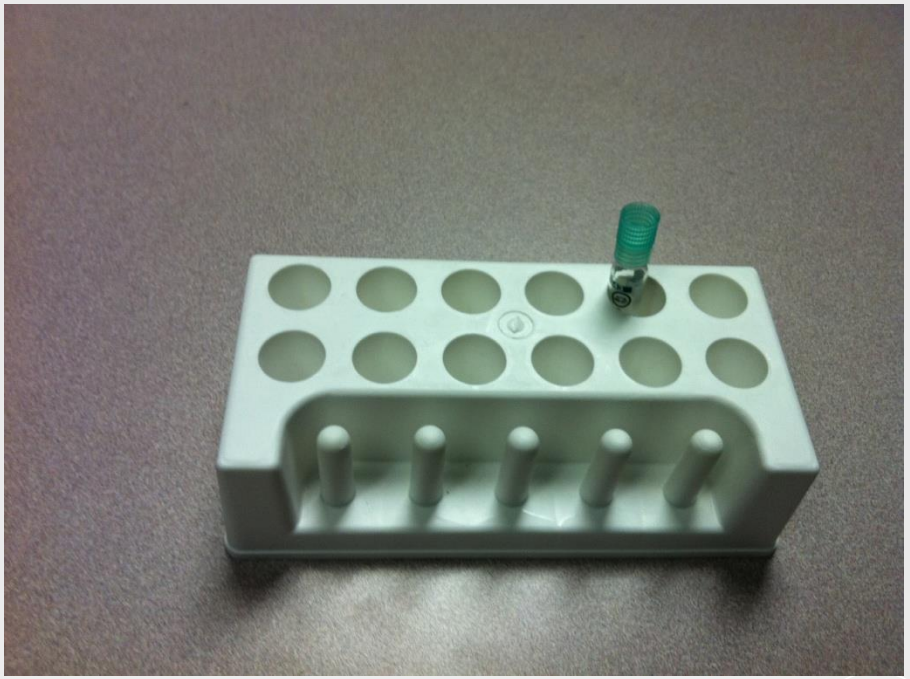
TNTplus Ultra High Range 2 - 20 mg/L PO_4-P, Method 10210 (TNT845)

After the timer expires carefully remove the hot vial or vials form the reactor. Insert vial or vials in a test tube rack and allow to cool to room temperature.



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TNTplus Ultra High Range 2 - 20 mg/L PO_4-P, Method 10210 (TNT845)

Pipet 0.5 mL of Reagent B into the cooled vial

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TNTplus Ultra High Range 2 - 20 mg/L PO_4-P, Method 10210 (TNT845)

Screw a grey DosiCap C onto the vial

Invert the capped vial 2-3 times to dissolve the reagent in the DosiCap
Wait 10 minutes.

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TNTplus Ultra High Range 2 - 20 mg/L PO_4-P, Method 10210 (TNT845)

When the timer expires invert again 2-3 times and then insert into a Spectrophotometer for readings

Record the readings

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b. HACH Method TNT 843

HACH® TNTplus™

Storage +15°C...+25°C

TNT 843

Phosphorus Total
Phosphorus Reactive (ortho)

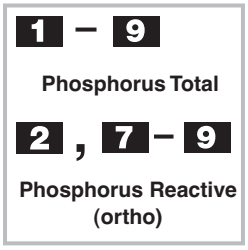
pH of sample: 2 – 10

Temperature of sample/reagent: 15 – 25°C

0.05 – 1.50 mg/L PO_4-P
0.15 – 4.50 mg/L PO_4
Low Range

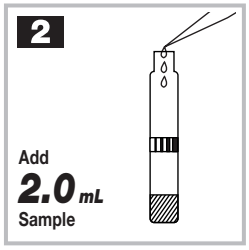
Special Notes (For more detailed information: HACH Procedure Manual)

- Please read ***Safety Advice*** and ***Expiration Date*** on package.
- **Range of application:** For wastewater, drinking water, boiler water, surface water and process analysis
- If test is not performed at the ***recommended temperature*** an ***incorrect*** result may be obtained.
- A blue color will develop if phosphorus is present.
- **DR 1900:** Select Program **843**.

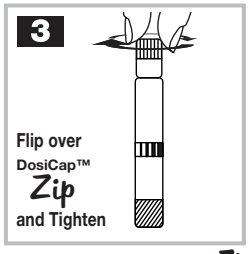


For Phosphorus Total perform steps **1 – 9**.
For Phosphorus Reactive (ortho) perform steps **2** and **7 – 9**.

Carefully remove the foil from the ***DosiCap™ Zip*** and unscrew cap.

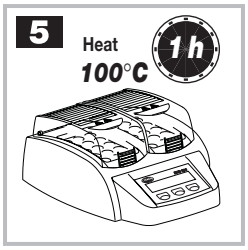


Pipet ***2.0 mL*** of sample into the vial.

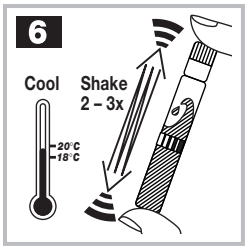


Screw the ***DosiCap™ Zip*** back on.

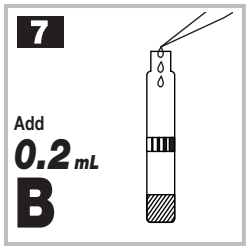
Shake ***firmly*** 2 – 3 times.



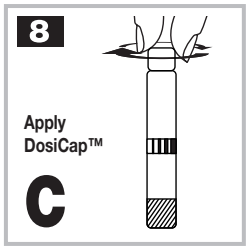
Heat ***one hour*** at 100°C in the reactor.



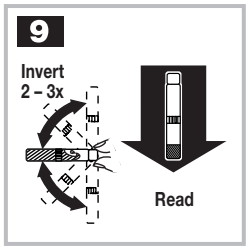
Shake the ***cooled*** vial ***firmly*** 2 – 3 times.



Pipet into the vial:
0.2 mL Reagent ***B***.
Close Reagent B ***immediately*** after use.



Screw a ***grey DosiCap™ C*** onto the vial.



Invert 2 – 3 times. After ***10 minutes*** invert again 2 – 3 times.
Thoroughly clean the outside of the vial and insert it into the photometer.
The ***barcode*** is identified, an ***automatic evaluation*** is carried out after the vial is inserted.

Principle	**Interferences**		
Phosphate ions react with molybdate and antimony ions in an acidic solution to form an antimonyl phosphomolybdate complex, which is reduced by ascorbic acid to phosphomolybdenum blue.	The ions listed below have been individually checked up to the given concentrations and do not cause interference. We have not determined cumulative effects and the influence of other ions. Measurement results can be verified using sample dilutions or standard additions.		
	5000 mg/L: SO_4^{2-}	**250 mg/L:** Ca^{2+}	**5 mg/L:** Sn^{4+}, Hg^{2+}
	2000 mg/L: Cl^-	**100 mg/L:** Mg^{2+}	**2.5 mg/L:** Ag^+, Pb^{2+}
	1000 mg/L: K^+ , Na^+	**50 mg/L:** Co^{2+}, Fe^{2+}, Fe^{3+}, Zn^{2+}, Cu^{2+}, Ni^{2+}, I^-, NO_2^-, Cd^{2+}, NH_4^+, Mn^{2+}, Al^{3+}, CO_3^{2-}, SiO_2	**1 mg/L:** Cr^{3+}
	500 mg/L: NO_3^-		**0.5 mg/L:** Cr^{6+}

Note: *For more detailed information see the HACH Procedure Manual.*

TNT 843

Phosphore total
Phosphore réactif

pH d'échantillon : 2 – 10

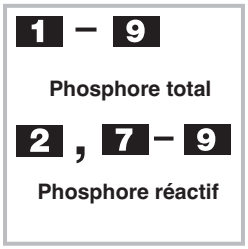
Température d'échantillon/réactif : 15 – 25°C

0.05 – 1.50 mg/L PO_4-P
0.15 – 4.50 mg/L PO_4
Gamme Basse

Stabilité

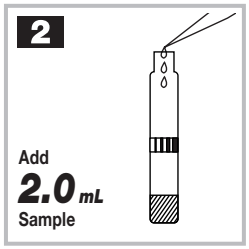
Remarque importante (Plus d'informations: Manuel des Procédures HACH)

- Lire s.v.p. les ***conseils de sécurité*** et la ***date de péremption*** comme indiqués sur l'emballage.
- **Validité et application(s) :** eaux de rejet, eaux potables, eaux de chaudière, eaux de surface et analyses en mode continu
- Des ***températures différentes*** influencent ***l'exactitude*** des résultats.
- Une coloration bleue apparaîtra en cas de présence de phosphore.
- **DR 1900:** Sélectionnez Programme **843**.

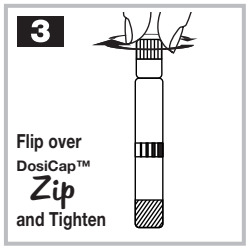


Pour le phosphore total exécutez les étapes **1 – 9**. Pour le phosphore réactif exécutez l'étape **2** et les étapes **7 – 9**.

Enlevez ***délicatement*** la feuille de protection du ***DosiCap™ Zip*** détachable.

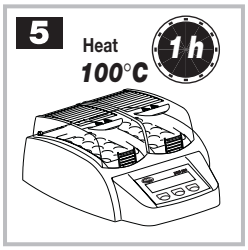


Pipetter ***2.0 mL*** d'échantillon.

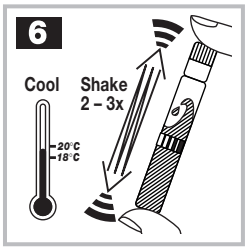


Vissez le ***DosiCap™ Zip*** ; dirigeant le cannelage vers le haut.

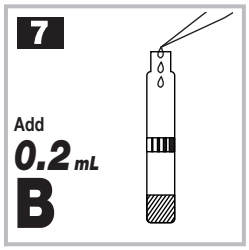
Secouer ***énergiquement***.



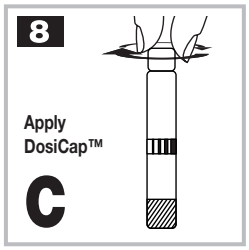
Chauffer ***une heure*** à 100°C dans le thermostat



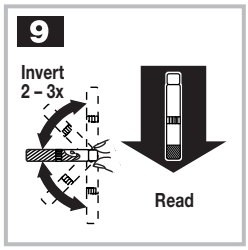
Secouer ***énergiquement*** la cuve une fois ***refroidie***.



Pipetter dans la cuve : ***0.2 mL*** de réactif ***B***. Fermer ***immédiatement*** le réactif B après emploi.



Visser un ***DosiCap™ C gris*** sur la cuve.



Mélanger le contenu de la cuve en la retournant 2 – 3 fois de suite. Attendre ***10 minutes***, retourner la cuve de nouveau 2 – 3 fois.
Bien nettoyer l'extérieur de la cuve et l'introduire dans le compartiment pour cuve du photomètre.
Le ***code à barres*** est identifié, une ***évaluation automatique*** est réalisée après l'insertion de la cuve.

Principe	**Interférences**		
Les ions phosphate réagissent en solution acide avec les ions molybdate et antimoine pour donner un complexe de phosphore molybdate d'antimoine. Celui-ci est réduit par l'acide ascorbique en bleu de phosphoremolybdène.	Les ions mentionnés en bas ont été vérifiés séparément, ils n'interferent pas jusqu'aux concentrations indiquées. Nous n'avons cependant pas étudié l'effet cumulatif et l'influence d'ions supplémentaires. Les résultats de mesures sont à vérifier par un contrôle de plausibilité (dilution et/ou addition).		
	5000 mg/L: SO_4^{2-}	**250 mg/L:** Ca^{2+}	**5 mg/L:** Sn^{4+}, Hg^{2+}
	2000 mg/L: Cl^-	**100 mg/L:** Mg^{2+}	**2.5 mg/L:** Ag^+, Pb^{2+}
	1000 mg/L: K^+ , Na^+	**50 mg/L:** Co^{2+}, Fe^{2+}, Fe^{3+}, Zn^{2+}, Cu^{2+}, Ni^{2+}, I^-, NO_2^-, Cd^{2+}, NH_4^+, Mn^{2+}, Al^{3+}, CO_3^{2-}, SiO_2	**1 mg/L:** Cr^{3+}
	500 mg/L: NO_3^-		**0.5 mg/L:** Cr^{6+}

Note: *Pour plus d'informations voir le Manuel de Procédures HACH.*

HACH® TNTplus™

Almacenamiento +15°C – +25°C

TNT 843

Fósforo total
Fósforo reactivo

pH de la muestra: 2 – 10

Temperatura de la muestra/reactivo: 15 – 25°C

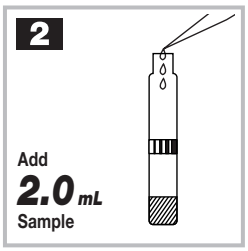
0.05 – 1.50 mg/L PO_4-P
0.15 – 4.50 mg/L PO_4
Rango Bajo

Notas especiales (Para más información: Manual de Procedimientos de HACH)

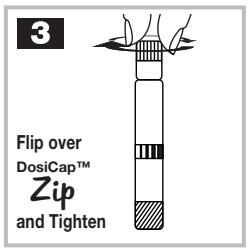
- Leer las ***Indicaciones de Seguridad*** y la ***Fecha de Caducidad*** en el envase.
- **Campo de aplicación:** Para aguas superficiales, agua potable, agua de calderas, aguas residuales y analítica de procesos
- Dependencia de la temperatura: En caso ***contrario***, pueden obtenerse resultados ***incorrectos***.
- En presencia de fosfato, aparecerá un color azul.
- **DR 1900**: Seleccionar programa **843**.

Para Fósforo total realizar los pasos **1 – 9** y, para Fósforo reactivo, los pasos **2** y **7 – 9**.

Retirar con ***sumo cuidado*** el precinto de papel de aluminio del ***DosiCap™ Zip*** roscado.

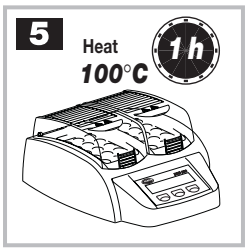


Pipetear ***2.0 mL*** de muestra.

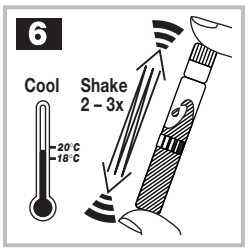


Roscar el ***DosiCap™ Zip***, estría hacia arriba.

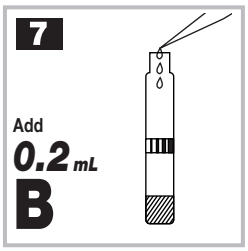
Agitar ***enérgicamente***.



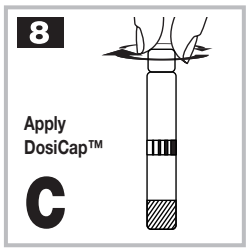
Calentar durante ***una hora*** a 100°C en el termostato.



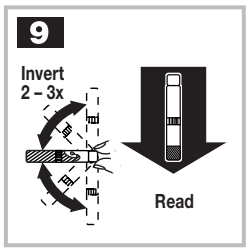
Agitar ***enérgicamente*** la cubeta enfriada.



Pipetear en la cubeta: ***0.2 mL*** de reactivo ***B***. Cerrar el reactivo B ***inmediatamente*** después del uso.



Roscar un ***DosiCap™ C*** de color ***gris*** sobre la cubeta.



Agitar la cubeta 2 – 3 veces. Transcurridos ***10 minutos*** volver a agitar la cubeta 2 – 3 veces. Limpiar bien el exterior de la cubeta y colocarla en el soporte portacubetas. Después de colocar la cubeta, el ***código de barras*** es identificado y se lleva a acabo una ***evaluación automática***.

Principio	**Interferencias**		
Los iones fosfato reaccionan en solución ácida con iones molibdato y antimonio formando un complejo antimonilfosfomolibdato que, mediante ácido ascórbico, se reduce a azul de fosfomolibdeno.	Los iones relacionados abajo han sido comprobados individualmente hasta las concentraciones indicadas y no causan interferencias. No hemos determinado el efecto acumulativo; ni la influencia de otros iones. Los resultados de medición han de ser sometidos a un control de verosimilitud (diluir y/o adicionar).		
	5000 mg/L: SO_4^{2-}	**250 mg/L:** Ca^{2+}	**5 mg/L:** Sn^{4+}, Hg^{2+}
	2000 mg/L: Cl^-	**100 mg/L:** Mg^{2+}	**2.5 mg/L:** Ag^+, Pb^{2+}
	1000 mg/L: K^+ , Na^+	**50 mg/L:** Co^{2+}, Fe^{2+}, Fe^{3+}, Zn^{2+}, Cu^{2+}, Ni^{2+}, I^-, NO_2^-, Cd^{2+}, NH_4^+, Mn^{2+}, Al^{3+}, CO_3^{2-}, SiO_2	**1 mg/L:** Cr^{3+}
	500 mg/L: NO_3^-		**0.5 mg/L:** Cr^{6+}

Nota: *Para más información, véase el Manual de Procedimientos de HACH.*

Datatable/Table des données/Tabla de datos DR 5000/DR 6000	
Program/Progamme/Programa	843
Name/Nom/Nombre	Phosphate/Fosfato
Variables	
F1	4.997
F2	0.561
λ1	850 nm
No	6
More detailed information for editing a Barcode Test see the HACH User Manual DR 5000/DR 6000.	
Pour des informations détaillées concernant la mise à jour/ modification des Tests des codes à barres voir le mode d'emploi HACH DR 5000/DR 6000.	
Para más información detallada sobre la actualización/ edición de test de códiges de barras, véase el Manual del Usario de HACH DR 5000/DR 6000.	

Datatable/Table des données/Tabla de datos DR 2800/DR 3800/DR 3900	
Program/Progamme/Programa	843
Name/Nom/Nombre	Phosphate/Fosfato
Variables	
F1	4.334
F2	0.554
λ1	890 nm
No	6
More detailed information for editing a Barcode Test see the HACH User Manual DR 2800/DR 3800/DR 3900.	
Pour des informations détaillées concernant la mise à jour/ modification des Tests des codes à barres voir le mode d'emploi HACH DR 2800/DR 3800/DR 3900.	
Para más información detallada sobre la actualización/ edición de test de códiges de barras, véase el Manual del Usario de HACH DR 2800/DR 3800/DR 3900.	

FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:
In the U.S.A. – **Call toll-free 800-227-4224**
Outside the U.S.A. – **Contact the HACH office or distributor serving you.**
On the Worldwide Web – **www.hach.com**; E-mail – **techhelp@hach.com**

HACH COMPANY
WORLD HEADQUARTERS
Telephone: (970) 669-3050
FAX: (970) 669-2932

SEP 14 3ed

c. HACH Method TNT 843, 844, 845

Hach Company TNTplus™ Phosphorus – Spectrophotometric Measurement of Phosphorus in Water and Wastewater

Hach Company TNTplus™ Phosphorus

Method 10209/10210/843/844/845
January 2008

Spectrophotometric Measurement of ortho - and Total Phosphorus in Water and Wastewater

1.0 Scope and Application

1.1 These procedures cover the determination of specified forms of phosphorus in drinking water, surface and saline waters, domestic and industrial wastes.

1.2 The procedures are based on reactions that are specific for the orthophosphate ion. Thus, depending on the prescribed pre-treatment of the sample, the various forms of phosphorus given may be determined. These forms are defined in Section 4.0.

1.3 The method is applicable in the range from 0.01 to 20 mg P /L, depending on the test range of the TNTplus Phosphorus kit.

2.0 Summary of Method

2.1 Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of phosphorus to form an antimony-phospho-molybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the phosphorus concentration and is measured at 650 or 880 ± 5 nm.

2.2 Only orthophosphate forms a blue color in this test. Polyphosphates (and some organic phosphorus compounds) may be converted to the orthophosphate form by sulfuric acid hydrolysis. Organic phosphorus compounds may be converted to the orthophosphate form by persulfate digestion.

3.0 Interferences

3.1 There are no interferences caused by copper, iron, or silicate at concentrations many times greater than their reported concentration in seawater. However, high iron concentrations can cause precipitation of and subsequent loss of phosphorus.

3.2 The salt error for samples ranging from 5 to 20% salt content was found to be less than 1%.

3.3 Arsenate is determined similarly to phosphorus and should be considered when present in concentrations higher than phosphorus. However, at concentrations found in seawater, it does not interfere.

4.0 Definitions

4.1 Total Phosphorus (P)--all of the phosphorus present in the sample, regardless of form, as measured by the persulfate digestion procedure.

4.1.1 Total Orthophosphate (P, ortho)--inorganic phosphorus [$(PO_4\text{-})^{-3}$] in the sample as measured by the direct colorimetric analysis procedure.

4.1.3 Total Organic Phosphorus (P, org)--phosphorus (inorganic plus oxidizable organic) in the sample measured by the persulfate digestion procedure, and minus hydrolyzable phosphorus and orthophosphate.

4.2 Dissolved Phosphorus (P-D)--all of the phosphorus present in the filtrate of a sample filtered through a phosphorus-free filter of 0.45 micron pore size and measured by the persulfate digestion procedure.

4.2.1 Dissolved Orthophosphate (P - D, ortho)--as measured by the direct colorimetric analysis procedure.

4.2.2 Dissolved Organic Phosphorus (P - D, org)--as measured by the persulfate digestion procedure, and minus dissolved hydrolysable phosphorus and orthophosphate.

4.3 The following forms, when sufficient amounts of phosphorus are present in the sample to warrant such consideration, may be determined:

4.3.1 Insoluble Phosphorus (P - I) = (P) - (P - D).

4.3.1.1 Insoluble orthophosphate (P - I, ortho) = (P, ortho) - (P - D, ortho).

4.3.1.2 Insoluble Organic Phosphorus (P - I, org) = (P, org) - (P - D, org).

5.0 Safety

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest possible level. It is suggested that the laboratory perform personal hygiene monitoring of each analyst using this method and that the results of this monitoring be made available to the analyst.

5.2 Unknown samples may contain high concentrations of volatile toxic compounds. Sample containers should be opened in a hood and handled with gloves to prevent exposure.

5.3 This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of any chemicals specified in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses. Additional information on laboratory safety can be found in Sections 16.6 and 16.7.

6.0 Equipment

Note: *Brand names, suppliers, and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using apparatus and materials other than those specified here, but demonstration of equivalent performance that meets the requirements of this method is the responsibility of the laboratory.*

6.1 Sampling equipment

6.1.1 Sample collection bottles—Glass, approximately 1-L, with PTFE-lined screw cap.
Note: *In those instances necessitating collection of a smaller aliquot, a smaller sample container may be used.*

6.1.2 Cleaning

6.1.2.1 All glassware used should be washed with hot 1:1 HCl and rinsed with distilled water. Preferably, this glassware should be used only for the determination of phosphorus and after use it should be rinsed with distilled water and kept covered until needed again. If this is done, the treatment with 1:1 HCl is only occasionally required. Commercial detergents should never be used.

6.2 Equipment for glassware cleaning

6.2.2 Oven - Capable of maintaining a temperature within ± 5°C in the range of 100–250°C.

6.3 Equipment for sample analysis

6.3.1 Hach DR 6000, DR 5000, DR 3800, DR3900, or DR 2800 spectrophotometer.

6.3.2 DRB200 Digital Reactor Block for TNTplus: 30x13mm vial wells, 115 Vac (P/N DRB200-03)

6.4 Equipment for standard preparation

6.4.1 Volumetric flask – Glass, 1000-mL.

6.4.2 Volumetric flask – Glass, 50-mL.

6.4.3 Volumetric pipette – glass, assorted sizes.

7.0 Reagent and Standards

7.1 Reagent water – Water in which phosphorus is not detected at or above the method level of this method. Bottled distilled water, or water prepared by passage of tap water through ion exchange and activated carbon have been shown to be acceptable sources of reagent water.

7.2 Hach Company TNTplus Phosphorus Kits (TNT843, 0.01 – 1.5 mg P/L; TNT844, 0.5 – 5.0 mg P/L; TNTplus 845, 2.0 – 20 mg P/L; TNTplus 846, 1.6 – 30 mg P/L).

7.3 Hach Company Phosphate Standard Solution, 50 mg/L as PO_4, Cat. No. 171-49.

7.3.1 Prepare a secondary standard spiking solution by diluting 30.0 mL of standard solution (Section 7.3) to 1000 mL. Final concentration = 0.0005 mg P/mL.

7.4 Method detection limit solution

7.4.1 Prepare 7 or more replicate MDL solutions by diluting 1.0 mL of standard spiking solution (Section 7.3.1) to 50 mL. Final concentration = 0.01 mg P/L.

7.5 Initial precision and recovery solution

7.5.1 TNTplus 843, 0.01 – 1.5 mg P/L.

7.5.1.1 Prepare 4 or more replicate IPR solutions by diluting 10.0 mL of standard spiking solution (Section 7.3.1) to 50 mL. Final concentration = 0.10 mg P/L.

7.6 On-going precision and recovery

7.6.1 TNTplus 843, 0.01 – 1.5 mg P/L.

7.6.1.1 Prepare 1 or more solutions by diluting 1.0 mL of standard spiking solution (Section 7.3.1) to 50 mL. Final concentration = 0.10 mg P/L.

8.0 Sample Collection Preservation and Storage

8.1 If benthic deposits are present in the area being sampled, great care should be taken not to include these deposits.

8.2 Sample containers may be of plastic material, such as cubitainers, or of Pyrex glass.

8.3 If the analysis cannot be performed the day of collection, the sample should be preserved by the addition of 2 mL conc. H_2SO_4 per liter and refrigeration at 4°C.

9.0 Quality Control

9.1 It is recommended that each laboratory that uses this method be required to operate a formal quality assurance program (Section 16.1). The minimum requirements of this program consist of an initial demonstration of laboratory capability and ongoing analyses of laboratory prepared water standards as a test of continued performance to assess accuracy and precision. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1.1 The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.
The laboratory shall, on an ongoing basis, demonstrate through analysis of the ongoing precision and recovery sample that the analysis system is in control. This procedure is described in Sections 8.3.

9.1.2 Accompanying QC for the determination of P is required per analytical batch. An analytical batch is a set of samples processed during a contiguous 8-hour period. Each analytical batch must be accompanied by an ongoing precision and recovery sample, matrix spike sample, and matrix spike duplicate sample resulting in a minimum of four analyses (1 OPR, 1 sample, MS, and MSD).

9.2 Initial demonstration of laboratory capability.

9.2.1 To establish the ability to detect nitrite the analyst shall determine the MDL and ML per the procedure in 40 CFR 136, Appendix B (Section 16.5) using the apparatus, reagents, and standards that will be used in the practice of this method. An achieved MDL and ML less than or equal to the MDL in Section 13.0 is recommended prior to the practice of this method.

9.2.1 Prepare and measure seven replicates of the MDL standard according to the procedure beginning in Section 7.4.1.

9.2.2 Initial precision and recovery (IPR) - To establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations:

9.2.3.1 Prepare and measure four samples of the IPR standard according to the procedure beginning in Section 7.5.

9.2.3.2 Using the results of the set of four analyses, compute the average percent recovery (X) and the standard deviation of the percent recovery (*s*) for nitrite. Use the following equation for calculation of the standard deviation of the percent recovery:

$$s = \sqrt{\frac{\sum x^2 - \frac{\left(\sum x\right)^2}{n}}{n-1}}$$

where:

n = Number of samples
X = % recovery in each sample

9.2.3.3 Compare *s* and X with the corresponding limits for initial precision and recovery in Table 1. If *s* and X meet the acceptance criteria, system performance is acceptable and analysis of samples may begin. If, however, *s* exceeds the precision limit or X falls outside the range for recovery, system performance is unacceptable. In this event correct the problem, and repeat the test.

9.3 Ongoing precision and recovery - To demonstrate that the analysis system is in control, and acceptable precision and accuracy is being maintained with each analytical batch, the analyst shall perform the following operations:

9.3.1 Prepare a precision and recovery standard with each analytical batch according to the procedure beginning in Section 7.6.

9.3.2 At the end of each analytical batch of samples, analyze a precision and recovery standard and compare the concentration recovery with the limits for ongoing precision and recovery in Table 3. If the recovery is in the range specified, measurement process is in control and analysis of samples may proceed. If, however, the recovery is not in the specified range, the analytical process is not in control. In this event, correct the problem, re-analyze analytical batch, repeating the ongoing precision and recovery test.

9.3.3 The laboratory should add results that pass the specification in Section 13.0 to IPR and previous OPR data and update QC charts to form a graphic representation of continued laboratory performance. The laboratory should also develop a statement of laboratory data quality for each analyte by calculating the average percent recovery (R) and the standard deviation of the percent recovery (sr). Express the accuracy as a recovery interval from R - 2sr to R + 2sr. For example, if R = 95% and sr = 5%, the accuracy is 85% to 105%.

9.4 Depending upon specific program requirements, field replicates may be required to assess the precision and accuracy of the sampling and sample transporting techniques.

10.0 Calibration and Standardization

10.1 The Hach Company DR series spectrophotometers have a built-in calibration that is automatically used when the TNTplus Phosphorus sample vial is placed in the cell holder of the instrument. No further initial calibration is required. However, the instruments have the capability of developing a user-calibration. See manufacturer's manual for instructions.

11.0 Procedure

11.1 Instrument Setup – follow the instrument manufacturer's instructions for instrument setup.

11.2 Sample Preparation

11.2.1 Ortho-phosphate

11.2.1.1 Adjust sample pH to 2 – 10.

11.2.1.2 Sample temperature should be 15 – 25^{0}C.

11.2.1.3 Remove DosiCap^{TM} Zip from vial and remove aluminum foil seal.

11.2.1.4 Depending on TNTplus Phosphorus Test Kit, add designated amount of sample (see test kit instructions) to vial.

11.2.1.5 Attached DosiCap Zip (reagent side down) to vial.

11.2.1.6 Agitate sample in vial until reagent in DosiCap Zip has dissolved.

11.2.1.7 Allow the reagents and sample to react for 10 minutes.

11.2.1.8 Place test vial in spectrophotometer and read result.

11.2.2 Total Phosphate

11.2.2.1 Adjust sample pH to 2 – 10.

11.2.2.2 Sample temperature should be 15 – 25^{0}C.

11.2.2.3 Remove DosiCap^{TM} Zip from vial and remove aluminum foil seal.

11.2.2.4 Depending on TNTplus Phosphorus Test Kit, add designated amount of sample (see test kit instructions) to vial.

11.2.2.5 Attached DosiCap Zip (reagent side down) to vial.

11.2.2.6 Agitate sample in vial until reagent in DosiCap Zip has dissolved.

11.2.2.7 Place test vial in heating block and digest at 150^0C., for 15 minutes.

11.2.2.8 Cool test vial to room temperature, remove and discard DosiCap Zip, and add designated amount of Reagent B (see test kit instructions) to test vial.

11.2.2.9 Attach DosiCap from Bottle C.

11.2.2.10 Agitate sample in vial until reagent in DosiCap Zip has dissolved.

11.2.2.11 Allow the reagents and sample to react for 10 minutes.

11.2.2.12 Place test vial in spectrophotometer and read result.

12.0 Data Analysis and Calculations

12.1 Phosphorus concentration is calculated automatically against internal instrument calibration.

13.0 Method Performance

Acceptance Criterion	Section	Limit
Method Detection Limit	9.2.1	0.003 mg/L P
Minimum Limit	9.2.1	0.01 mg/L P
Initial Accuracy	9.2.2	88.6%
Initial Precision	9.2.2	0.44%
On-going Accuracy	9.3	98%

14.0 Pollution Prevention

14.1 Follow guidelines in Section 15.

15.0 Waste Management

15.1 It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect air, water, and land by minimizing and control all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

15.2 For further information on waste management, consult "The Waste Management manual for Laboratory Personnel", and Less is Better: "Laboratory Chemical Management for Waste Reduction", both available from the American Society's Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington, D.C. 20036.

16.0 References

16.1 "Handbook of Analytical Quality Control in Water and Wastewater Laboratories," USEPA, EMSL-CI, Cincinnati, OH 45268, EPA-600-4-79-019, March 1979.

16.2 Standard Methods for the Performance of Water and Wastewater, 20th Edition, p 443, Method 5210B (1998).

16.3 International Oceanographic Tables, Vol. 1, National Institute of Oceanography of Great Britain, Womley, Godaming, Surrey, England and Uncesco, Paris 1971.

16.4 40 CFR 136, Appendix A, Methods 1624 and 1625.

16.5 40 CFR 136, Appendix B.

16.6 "OSHA Safety and Health Standards, General Industry," (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976)

16.7 "Safety in Academic Chemistry Laboratories," American Chemical Society, Committee on Chemical Safety, 3rd Edition, 1979.

17.0 Tables

17.1 Acceptance Criteria for Performance tests – The QC performance criteria for this method was performed with a Hach Company DR 5000 spectrophotometer using Hach Company TNTplus Phosphorus Kit.

Table 1. Initial Precision and Recovery Method Performance

IPR Concentration	Average Recovery (%)	Rel. Standard Deviation (%)
0.10 mg/L P	88.6	0.44

Table 2. Method Detection Limit and Method Limit Performance

MDL Test Concentration	MDL	ML
0.01 mg P/L	0.003 mg P/L	0.01 mg P/L

Table 3. On-going Recovery Performance

OPR Concentration	Average % Recovery
0.10 mg P/L	98%

18.0 Glossary of Definitions and Purposes

The definitions and purposes are specified to this method but have been conformed to common usage as much as possible.

18.1 Units of weight and measure and their abbreviations

18.1.1 Symbols

°C degrees Celsius

18.1.2 Alphabetical characters

mg/L milligram per liter

18.2 Definitions, acronyms, and abbreviations

18.2.1 MDL: Method detection limit

18.2.2 ML: Method limit

18.2.3 IPR: Initial precision and recovery

18.2.4 OPR: On-going precision and recovery

18.2.5 MS: Matrix spike

Phosphorus
Method Comparison Tables

	Method 365.3 /SM 4500-P E	Hach TNTplus Phosphorus (10209/10210)
Scope and Application	0.01 – 0.50 mg P/L	0.01 – 20 mg P/L
Summary of Method	Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of phosphorus to form an antimony-phospho-molybdate complex. The complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the phosphorus concentration. Only orthophosphate forms a blue color in this test. Organic phosphorus compounds may be converted to the orthophosphate form by persulphate digestion.	Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of phosphorus to form an antimony-phospho-molybdate complex. The complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the phosphorus concentration. Only orthophosphate forms a blue color in this test. Organic phosphorus compounds may be converted to the orthophosphate form by persulphate digestion.
Interference	No interference is caused by copper, iron, or silicate at concentrations many times greater than their reported concentration in seawater. However, high iron concentrations can cause precipitation of and subsequent loss of phosphorus.	No interference is caused by copper, iron, or silicate at concentrations many times greater than their reported concentration in sea water. However, high iron concentrations can cause precipitation of and subsequent loss of phosphorus.
Equipment	Spectrophotometer	Spectrophotometer
Sample Handling/ Preservation	If the analysis cannot be performed the day of collection, the sample should be preserved by the addition of 2 mL conc. H_2SO_4.	If the analysis cannot be performed the day of collection, the sample should be preserved by the addition of 2 mL conc. H_2SO_4.
Reagents and Standards	Sulfuric Acid (source of acidity) Antimony potassium tartrate (complexing reagent) Ammonium molybdate (complexing reagent) Ascorbic acid (reducing reagent)) Persulfate (digestion reagent)	Sulfuric Acid (source of acidity) Antimony potassium tartrate (complexing reagent) Ammonium molybdate (complexing reagent) Ascorbic acid (reducing reagent) Lithium sulfate (stabilizer) Sulfamic acid (interference reagent) Tartaric acid (solubility enhancer) Persulfate (digestion reagent)
Method Performance	Precision and Accuracy: Orthophosphate <u>Conc. mg P/L</u> <u>Stdev.</u> <u>% Bias</u> 0.29 0.10 - 4.95 0.038 0.008 - 6.00 0.335 0.018 - 2.75 0.383 0.023 - 1.76 Organic Phosphate (Total Phosphate) 0.110 0.033 + 0.003 0.132 0.051 + 0.016 0.772 0.130 + 0.023 0.882 0.128 - 0.008	Precision and Accuracy: Orthophosphate Initial Precision and Recovery 99% (0.10 mg P/L Stdev. 0.86 Ongoing Precision and Recovery – 97% Effluent #1 (Loveland, CO) Average Matrix Spike Recovery – 99% Stdev – 0.51 (0.20 mg P/L spike) Effluent #2 (Boston, MA) Average Matrix Spike Recovery – 94% Stdev – 2.6 (0.20 mg P/L spike)

		Effluent #3 (Ventura, CA Average Matrix Spike Recovery – 99% Stdev – 0.01 (0.20 mg P/L spike) Method Detection Limit – 0.003 P/L @ (0.01 mg P/L spike) Organic Phosphate (Total Phosphate) Conc. mg P/L % Recovery % Bias 0.03 97 - 3 0.05 98 - 2 0.10 104 + 4 0.20 102 + 2 0.30 100 0 0.40 99 - 1 0.50 97 - 3 1.00 98 - 2 1.20 98 - 2 1.30 98 - 2 1.40 98 - 2 1.50 99 - 1 Effluent #1 (Loveland, CO) Average Matrix Spike Recovery – 98% Stdev – 1.17 (0.20 mg P/L spike) Effluent #2 (Boston, MA) Average Matrix Spike Recovery – 99% Stdev – 2.3 (0.20 mg P/L spike) Effluent #3 (Ventura, CA Average Matrix Spike Recovery – 101% Stdev – 0.29 (0.20 mg P/L spike) Method Detection Limit – 0.003 mg P/L (0.01 mg P/L spike)

5. TOTAL NITROGEN

a. Testing Procedures

Testing Methods – Total Nitrogen

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Testing Procedures

Total Nitrogen

Test ‘N Tube Low Range 0.5 - 25.0 mg/L N, Method 10071

TNTplus Low Range 1 - 16 mg/L N, Method 10208 (TNT 826)

TNTplus High Range 5 - 40 mg/L N, Method 10208 (TNT 827)

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INGHAM COUNTY
CLEAN WATER
DRAIN COMMISSIONER
PATRICK E. LINDEMANN

Test ‘N Tube Low Range 0.5 - 25.0 mg/L N

Nitrogen, Total

DOC316.53.001086

Persulfate Digestion Method — **Method 10071**

LR (0.5 to 25.0 mg/L N) — **Test ‘N Tube™ Vials**

Scope and Application: For water and wastewater

Test preparation

How to use instrument-specific information

The *Instrument-specific information* table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

Table 1 Instrument-specific information

Instrument	Light shield
DR 3900	LZV849
DR 3800, DR 2800, DR 2700	LZV646

Before starting the test:

DR 3900, DR 3800, DR 2800 and DR 2700: Install the light shield in Cell Compartment #2 before performing this test.
Digestion is required for determining total nitrogen.
This test is technique-sensitive. Invert the vials as described here to avoid low results: Hold the vial in a vertical position with the cap pointing up. Turn the vial upside-down. Wait for all of the solution to flow down to the cap. Pause. Return the vial to an upright position. Wait for all the solution to flow to the bottom of the vial. This process equals one inversion.
If the test overranges, repeat the digestion and measurement with diluted sample. The digestion must be repeated for accurate results.
Use the deionized water provided in the reagent set or Organic-free Water to prepare the standards and perform the procedure.

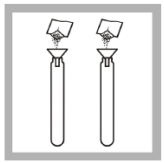
Collect the following items:

Description	Quantity
Test ‘N Tube™ LR Total Nitrogen Reagent Set	1
DRB200 Reactor	1
Funnel, micro	1
Light Shield or adapter (see *Instrument-specific information*)	1
Pipet, TenSette®, 1.0 to 10.0 mL plus tips	1
Test Tube Cooling Rack	1–3
Finger Cots	2

See *Consumables and replacement items* for reorder information.

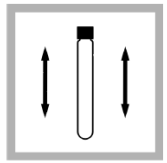
Persulfate digestion method

1. Turn on the DRB200 Reactor and heat to 105 °C.



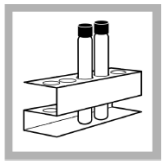
2. Using a funnel, add the contents of one Total Nitrogen Persulfate Reagent Powder Pillow to each of two Total Nitrogen Hydroxide Digestion Reagent vials. Wipe off any reagent that may get on the lip or the tube threads.
Note: *One reagent blank is sufficient for each set of samples.*

3. **Prepared Sample:** Add 2 mL of sample to one vial.
Blank Preparation: Add 2 mL of the deionized water included in the kit to a second vial.
Use only water that is free of all nitrogen-containing species as a substitute for the provided deionized water.

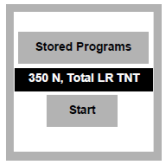


4. Cap both vials. Shake vigorously for at least 30 seconds to mix. The persulfate reagent may not dissolve completely after shaking. This will not affect accuracy.

5. Insert the vials in the reactor and close the lid. **Heat for exactly 30 minutes.**



6. Using finger cots, **immediately** remove the hot vials from the reactor. Cool the vials to room temperature.

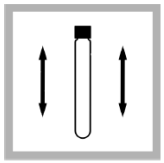


7. Select the test.
Insert an adapter if required (see *Instrument-specific information*).

8. Remove the caps from the digested vials and add the contents of one Total Nitrogen (TN) Reagent A Powder Pillow to each vial.

Test ‘N Tube Low Range 0.5 - 25.0 mg/L N

Persulfate digestion method (continued)

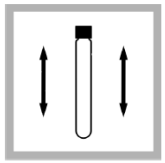


9. Cap the tubes and shake for 15 seconds.

10. Start the instrument timer.

A three-minute reaction period will begin.

11. After the timer expires, remove the caps from the vials and add one TN Reagent B Powder Pillow to each vial.

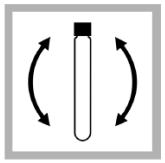


12. Cap the tubes and shake for 15 seconds. The reagent will not completely dissolve. This will not affect accuracy. The solution will begin to turn yellow.

13. Start the instrument timer.

A two-minute reaction period will begin.

14. Prepared Sample: After the timer expires, remove the caps from two TN Reagent C vials and add 2 mL of digested, treated sample to one vial. **Blank:** Add 2 mL of digested, treated reagent blank to the second TN Reagent C vial.



15. Cap the vials and invert ten times to mix. Use slow, deliberate inversions for complete recovery.

The tubes will be warm to the touch.

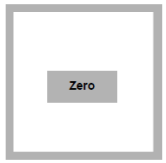
16. Start the instrument timer.

A five-minute reaction period will begin.

The yellow color will intensify.

Persulfate digestion method (continued)

17. Wipe the reagent blank and insert it into the 16-mm round cell holder.



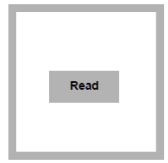
18. ZERO the instrument.

The display will show:

0.0 mg/L N

19. Wipe the reagent vial and insert it into the 16-mm round cell holder.

Note: *Multiple samples may be read after zeroing on one reagent blank.*



20. READ the results in mg/L N.

Blanks for colorimetric measurement

The reagent blank may be used up to seven days for measurements using the same lots of reagents. Store it in the dark at room temperature (18–25 °C). If a small amount of white floc appears within a week, discard the reagent blank and prepare a new one.

Interferences

The *Non-interfering substances* table shows substances that have been tested and found not to interfere up to the indicated levels (in mg/L). Interfering substances that resulted in a concentration change of ±10% appear in the *Interfering substances* table.

Table 2 Non-interfering substances

Interfering substance	Interference level
Barium	2.6 mg/L
Calcium	300 mg/L
Chromium ($^{3+}$)	0.5 mg/L
Iron	2 mg/L
Lead	6.6 µg/L
Magnesium	500 mg/L
Organic Carbon	150 mg/L
Phosphorus	100 mg/L
Silica	150 mg/L
Silver	0.9 mg/L
Tin	1.5 mg/L

Table 3 Interfering substances

Interfering substance	Interference level
Bromide	> 60 mg/L; positive interference
Chloride	> 1000 mg/L; positive interference

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Test ‘N Tube Low Range 0.5 - 25.0 mg/L N

This test performed with standard nitrogen solutions prepared from the following compounds obtained 95% recovery:

- Ammonium chloride
- Ammonium sulfate
- Ammonium acetate
- Urea
- Glycine

Ammonium chloride or nicotinic-PTSA spikes in domestic influent, effluent and the ASTM standard specification for substitute wastewater (D 5905-96) also resulted in i 95% recovery.

The large amounts of nitrogen-free organic compounds in some samples may decrease digestion efficiency by consuming some of the persulfate reagent. Samples known to contain high levels of organics should be diluted and re-run to verify digestion efficiency.

Sample collection, storage and preservation

- Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis.
- Preserve the sample by reducing the pH to 2 or less with concentrated (at least 2 mL/L) Sulfuric Acid.
- Store samples at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days.
- Warm stored samples to room temperature and neutralize with 5 N Sodium Hydroxide before analysis.
- Correct the test result for volume additions.

Accuracy check

This method generally yields 95–100% recovery on organic nitrogen standards. For proof of accuracy use Primary Standards for Kjeldahl Nitrogen.

1. Prepare one or more of the following three solutions. Each preparation is for an equivalent 25-mg/L N standard. Use the deionized water included in the kit or water that is free of all organic and nitrogen-containing species.
   a. Weigh 0.3379 g of Ammonium p-Toluenesulfonate (PTSA). Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.
   b. Weigh 0.4416 g of Glycine p-Toluenesulfonate (PTSA). Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.
   c. Weigh 0.5274 g of Nicotinic p-Toluenesulfonate (PTSA). Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.
2. Analyze each of these solutions using the test procedure above. Calculate the percent recovery for each using this formula. Refer to the *Percent recovery* table for more information.

$$\% \text{ recovery} = \frac{\text{measured concentration}}{25} \text{ i } 100$$

Refer to the *Percent recovery* table.

Table 4 Percent recovery

Compound	Lowest Expected % Recovery
Ammonia-PTSA	95%

Table 4 Percent recovery (continued)

Compound	Lowest Expected % Recovery
Glycine-PTSA	95%
Nicotinic-PTSA	95%

Analysts have found Ammonia-PTSA to be the most difficult to digest. Other compounds may yield different percent recoveries.

Standard additions method (sample spike)
Required for accuracy check:

- Ammonia Nitrogen Standard Solution, 1000-mg/L as NH_3–N
- Ampule breaker
- TenSette Pipet and tips
- Mixing cylinders (3)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Select standard additions from the instrument menu: **OPTIONS>MORE>STANDARD ADDITIONS**.
3. Accept the default values for standard concentration, sample volume and spike volumes. After the values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
4. Use the TenSette Pipet to prepare spiked samples: add 0.1 mL, 0.2 mL and 0.3 mL of standard to three 50-mL portions of fresh sample.
5. Follow the *Persulfate digestion method* test procedure for each of the spiked samples, starting with the 0.1 mL sample spike. Measure each of the spiked samples in the instrument.
6. Select **GRAPH** to view the results. Select **IDEAL LINE** (or best-fit) to compare the standard addition results to the theoretical 100% recovery.

Standard solution method
Note: *Refer to the instrument user manual for specific software navigation instructions.*

Required for accuracy check:

- 10-mg/L ammonia nitrogen standard solution

1. Substitute 2 mL of a 10-mg/L ammonia nitrogen standard solution in place of the sample. Follow the *Persulfate digestion method* test procedure.
2. To adjust the calibration curve using the reading obtained with the standard solution, navigate to Standard Adjust in the software: **OPTIONS>MORE>STANDARD ADJUST**.
3. Turn on the Standard Adjust feature and accept the displayed concentration. If an alternate concentration is used, enter the concentration and adjust the curve to that value.

Method performance

Program	Standard	Precision—95% Confidence Limits of Distribution	Sensitivity—Concentration per 0.010 Abs
350	10 mg/L NH_3–N	9.6–10.4 mg/L N	0.5 mg/L N

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Test ‘N Tube Low Range 0.5 - 25.0 mg/L N

Summary of method

An alkaline persulfate digestion converts all forms of nitrogen to nitrate. Sodium metabisulfite is added after the digestion to eliminate halogen oxide interferences. Nitrate then reacts with chromotropic acid under strongly acidic conditions to form a yellow complex with an absorbance maximum at 410 nm.

Consumables and replacement items

Required reagents

Description	Unit	Catalog number
Test 'N Tube™ Total Nitrogen Reagent Set, LR	50 vials	2672245

Required apparatus (powder pillows)

Description	Quantity	Unit	Catalog number
DRB200 Reactor, 110 V, 15x16 mm	1	each	LTV082.53.40001
OR			
DRB200 Reactor, 220 V, 15x16 mm	1	each	LTV082.52.40001
Funnel, micro	1	each	2584335
Pipet, TenSette®, 1.0 to 10.0 mL	1	each	1970010
Pipet Tips, for TenSette Pipet 19700-10	2	50/pkg	2199796
Test Tube Cooling Rack	1–3	each	1864100
Finger Cots	2	2/pkg	1464702

Recommended standards

Description	Unit	Catalog number
Ammonia Nitrogen Standard Solution, 1000-mg/L NH_3–N	1 L	2354153
Ammonia Nitrogen Standard Solution, 10-mg/L NH_3–N	500 mL	15349
Primary Standard Set, for Kjeldahl Nitrogen	set of 3	2277800
Wastewater Mixed Inorganic Standard for NH_3-H, NO_3-N, PO4, COD, SO_4,TOC	500 mL	2833149
Water, deionized	500 mL	27249
Water, organic-free	500 mL	2641549

Optional reagents and apparatus

Description	Unit	Catalog number
Balance, analytical, 80 g capacity, 115 VAC	each	2936701
Cylinder, mixing with stopper, 50 mL	each	2088641
Flask, volumetric, Class A, 1000 mL	each	1457453
Pipet, TenSette, 0.1 to 1.0 mL	each	1970001
Pipet Tips, for TenSette Pipet 1970001	50/pkg	2185696
Pipet tips for TenSette Pipet 1970001	1000/pkg	2185628
Pipet tips for TenSette Pipet 1970010	250-pkg	2199725
Sodium Hydroxide, 5 N	50 mL	245026

Nitrogen, Total

Optional reagents and apparatus

Description	Unit	Catalog number
Sulfuric Acid, concentrated	500 mL	97949
PourRite® Ampule breaker, 2-mL	each	2484600
Voluette® Ampule breaker 10 mL	each	2196800
Ammonia Nitrogen Standard Solution, 1-mg/L NH_3–N	500 mL	189149
Ammonia Nitrogen Standard Solution, 100-mg/L NH_3–N	500 mL	2406549
Ammonia Nitrogen Standard Solution, 2-mL PourRite Ampule, 50 mg/L	20/pkg	1479120
Ammonia Nitrogen Standard Solution, 10-mL Voluette Ampules, 10 mg/L	16/pkg	1479110
Ammonia Nitrogen Standard Solution, 10-mL Voluette Ampules, 150 mg/L	16/pkg	2128410

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FAX: (970) 669-2932

Edition 6

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Test ‘N Tube Low Range 0.5 - 25.0 mg/L N

- Turn on the DRB200 Reactor and heat to 105°C

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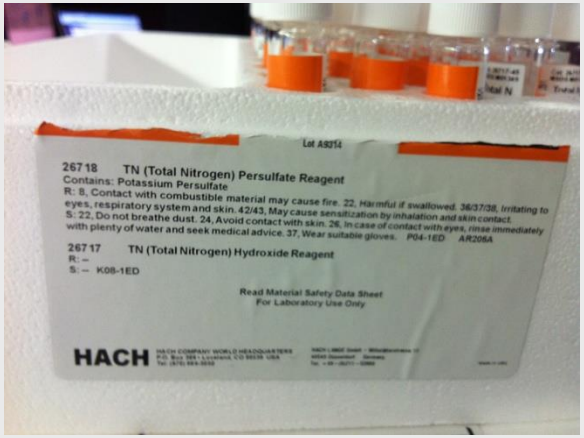

- Using a funnel, add the contents of one Total Nitrogen Persulfate Reagent Powder Pillow to each of two Total Nitrogen Hydroxide Digestion Reagent vials.
- Wipe off any reagent that may get on the lid or the tube threads
- One reagent blank is sufficient for each set of samples.

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- **Prepared Sample**: Add 2 mL of sample to one vial.
- **Blank Preparation**: Add 2 mL of the deionized water included in the kit to a second vial.
- Note: Use only water that is free of all nitrogen-containing species as a substitute for the provided deionized water.

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- Cap both vials.
- Shake vigorously for at least 30 seconds to mix.
- The persulfate reagent may not dissolve completely after shaking. This will not affect accuracy.

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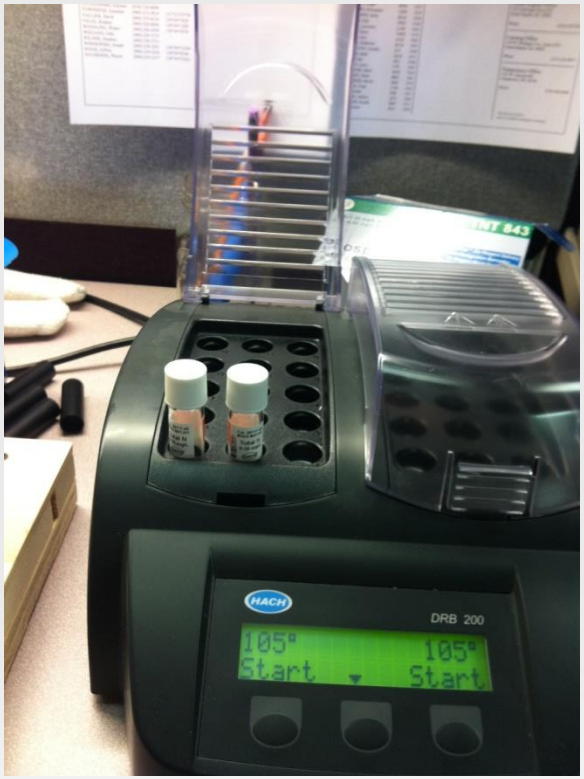

- Insert the vials in the reactor and close the lid.
- **Heat for exactly 30 minutes**.

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- Using finger cots, **immediately** remove the hot vials from the reactor.
- Cool the vials to room temperature.

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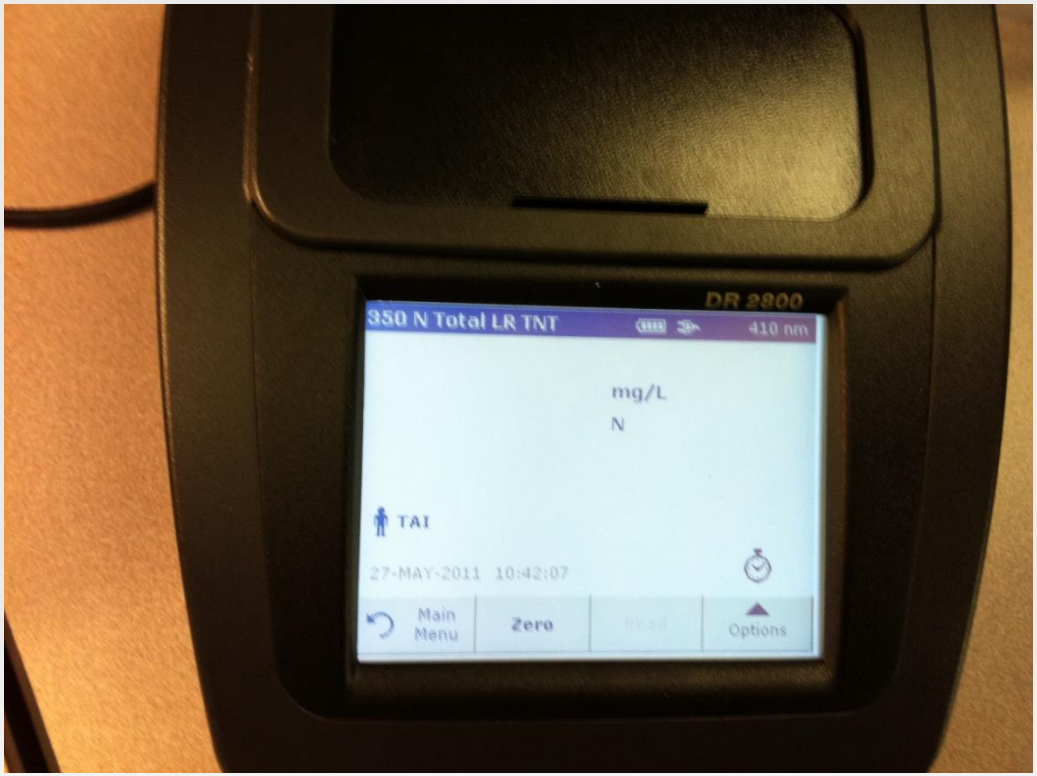

- Select the test 350 N, Total LR TNT

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- Remove the caps from the digested vials and add the contents of one Total Nitrogen (TN) Reagent A Powder Pillow to each vial.

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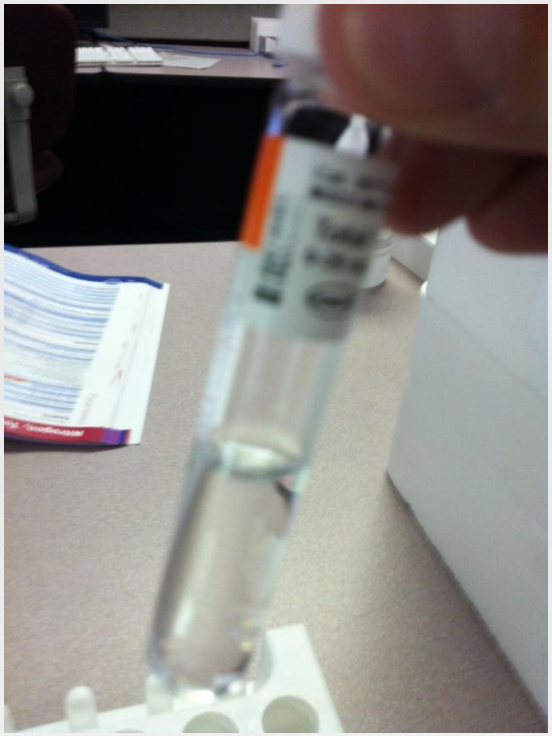

- Cap the tubes and shake for 15 seconds
- Start the instrument timer.
- A three-minute reaction period will begin.

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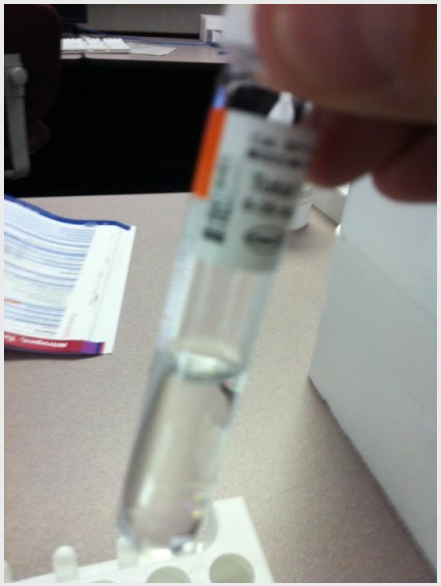
- After the timer expires, remove the caps from the vials and add one TN Reagent B Powder Pillow to each vial.

Test ‘N Tube Low Range 0.5 - 25.0 mg/L N



- Cap the tubes and shake for 15 seconds. The reagent will not completely dissolve.
- This will not affect accuracy. The solution will begin to turn yellow.
- Start the instrument timer. A two-minute reaction period will begin.

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Test ‘N Tube Low Range 0.5 - 25.0 mg/L N

- After the timer expires, remove the caps from two TN Reagent C vials and add 2 mL of digested, treated sample to one vial.
- Add 2 mL of digested, treated reagent blank to the second TN Reagent C vial.

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Test ‘N Tube Low Range 0.5 - 25.0 mg/L N

- Cap the vials and invert ten times to mix.
- Use slow, deliberate inversions for complete recover.
- Note: The tubes will be warm to the touch.
- Start instrument timer. A five-minute reaction period will begin. The yellow color will intensify.

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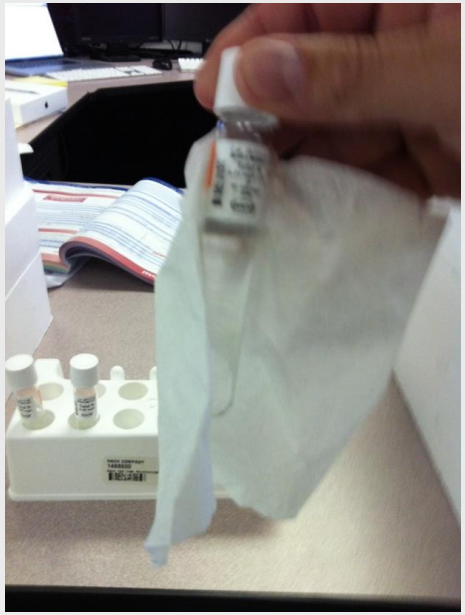

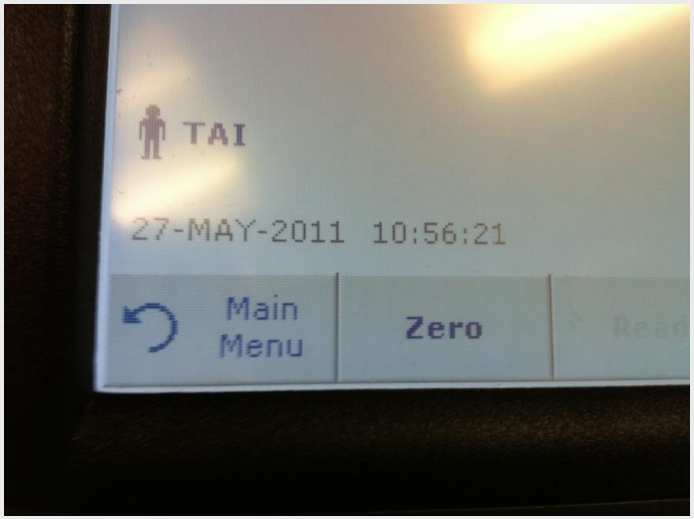

- Wipe the reagent blank and insert it into the 16-mm round cell holder.
- **Zero** the instrument. The display will show: 0.0 mg/L N

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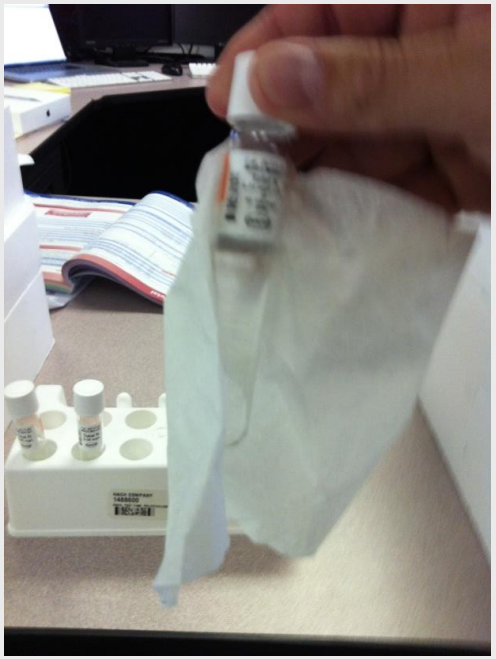

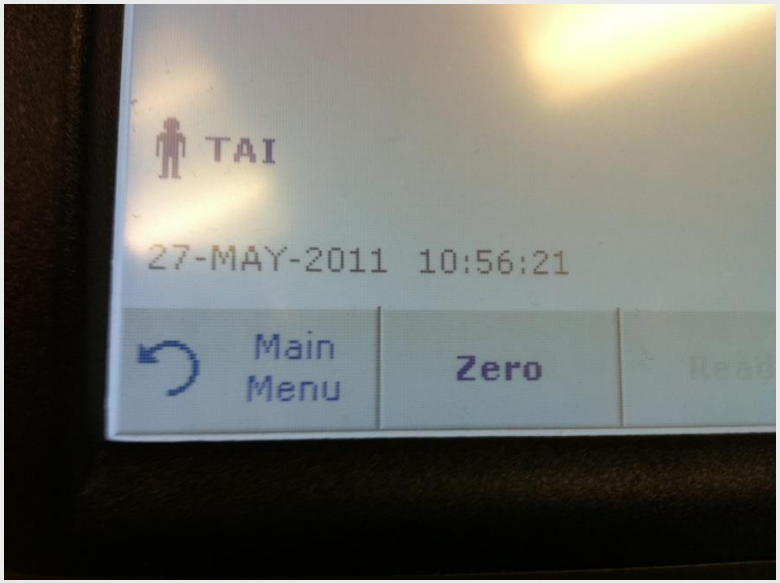

- Wipe the reagent vial and insert it into the 16-mm round cell holder.
- Note: Multiple samples may be read after zeroing on one reagent blank.
- **Read** the results in mg/L N.

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TNTplus Low Range 1 - 16 mg/L N

Nitrogen, Total

DOC316.53.01087

Persulfate Digestion Method — **Method 10208**

LR (1 to 16 mg/L N) — **TNTplus™ 826**

Scope and Application: For water and wastewater

Test preparation

How to use instrument-specific information

The *Instrument-specific information* table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

Table 1 Instrument-specific information

Instrument	Light shield
DR 3900	LZV849
DR 3800, DR 2800	LZV646

Before starting the test:

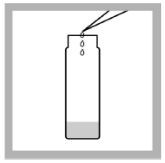
DR 3900, DR 3800, DR 2800: Install the light shield in Cell Compartment #2 before performing this test.
Read the Safety Advice and Expiration Date on the package.
Recommended sample and reagent temperature is 15–25 °C (59–77 °F). Recommended reagent storage temperature is 15–25 °C (59–77 °F).
Recommended sample pH is between 3–12.
Digestion is required for determining total nitrogen.
If the test is not performed at the recommended temperature an incorrect result may be obtained.
Use only high quality deionized water or Organic Free Water for preparing nitrogen standards or making sample dilutions and reagent blanks.
TNTplus methods are activated from the Main Menu when the sample vial is inserted into the sample cell holder.
IMPORTANT: Sodium hydroxide solution A / Oxidant tablet B / MicroCap C: After addition of reagents A, B and C the reagent bottles must be reclosed **immediately**.
IMPORTANT: Reaction Tubes (Ø 20 mm): Do not use reaction tubes more than **13 times**. After use, clean thoroughly with a brush and water, then rinse well with nitrogen-free distilled water and dry.
IMPORTANT: Turbidity: Slight turbidity does not interfere; high turbidity after the addition of the MicroCap C should be allowed to settle before pipetting the digested sample.

Collect the following items:

Description	Quantity
Nitrogen, Total, LR TNT826 Reagent Set	1
DRB200 Reactor, 20-mm wells	1
Light Shield (DR 3900, DR 3800, DR 2800)	1
Pipet for 1–5 mL volumes	1
Pipet Tips for 1–5 mL pipet	2
Pipet for 0.2–1.0 mL volumes	1
Pipet Tips for 0.2–1.0 mL pipet	2

Persulfate digestion method

1. Turn on the DRB200 Reactor and heat to 100 °C.

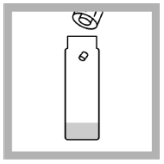


2. Add **1.3 mL** of sample, **1.3 mL** of Solution A and **1** Reagent B tablet in quick succession to a dry 20-mm reaction tube.

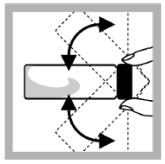
Close the reaction tube immediately. **Do not invert.**

3. Insert the reaction tubes in the reactor and close the lid. Heat for one hour.

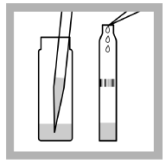
4. Remove the hot reaction tubes from the reactor. Cool the vials to room temperature (15–20 °C).



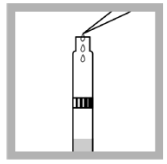
5. After the reaction tube has cooled, remove the cap and add 1 Micro Cap C to the tube.



6. Cap and invert the reaction tube 2–3 times until no more streaks can be seen in the reaction tube solution.



7. Pipet 0.5 mL (500 µL) of the digested sample from the reaction tube into a test vial.



8. Pipet 0.2 mL (200 µL) of Solution D into the test vial.

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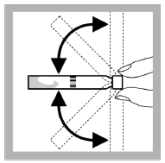
TNTplus Low Range 1 - 16 mg/L N

Persulfate digestion method (continued)



9. Quickly cap and invert the test vial 2–3 times until no more streaks can be seen in the vial solution.

10. Wait 15 minutes.

11. After the timer expires wipe the vial and Insert the prepared vial into the cell holder.

The instrument reads the barcode, then selects and performs the correct test.

Results are in mg/L N.

No instrument zero is required.

Reagent Blanks

A reagent blank can be measured and the value subtracted from the results of each test performed in same reagent lot. Use nitrogen-free deionized water in place of sample in the *Persulfate digestion method* test.

To subtract the value of the blank from a series of measurements:

1. Measure the blank per step 11.
2. Turn on the reagent blank option.
3. The measured value of the blank should be displayed in the highlighted box. Accept this value.

The reagent blank value will now be subtracted from all results until the function is turned off or a different method is selected. Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

Interferences

The ions listed in the *Interfering substances* table have been individually checked up to the given concentrations and do not cause interference. The cumulative effects of these ions or the influence of other ions have not been determined.

Table 2 Interfering substances

Interfering substance	Interference level
COD	400 mg/L
Chloride	800 mg/L

Sample collection, preservation and storage

- Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis.
- Preserve the sample by reducing the pH to 2 or less with concentrated (at least 2 mL/L) Sulfuric Acid.
- Store samples at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days.
- Warm stored samples to 15–25 °C and neutralize with 5 N Sodium Hydroxide before analysis.
- Correct the test result for volume additions.

Accuracy check

Standard solution method
Note: *Refer to the instrument user manual for specific software navigation instructions.*

Required for accuracy check:

- 10 mg/L ammonia nitrogen standard

  OR

- Wastewater Effluent Mixed Parameters Inorganics Standard

1. Use one of the following in place of the sample.
   - Use 1.3 mL of 10 mg/L standard in place of the sample in step 2.
   - Use 1.3 mL of a Wastewater Effluent Mixed Parameters Inorganics Standard in place of the sample in step 2. This standard contains 2 mg/L ammonia nitrogen and 4 mg/L nitrate nitrogen to give a combined standard of 6 mg/L as total nitrogen.
2. Follow the *Persulfate digestion method* test procedure.

Summary of method

Inorganically and organically bonded nitrogen is oxidized to nitrate by digestion with peroxodisulphate. The nitrate ions react with 2,6-dimethylphenol in a solution of sulphuric and phosphoric acid to form a nitrophenol. Test results are measured at 345 nm.

TNTplus Low Range 1 - 16 mg/L N

Consumables and replacement items

Required reagents

Description	Unit	Catalog number
Nitrogen Total, LR TNT826 Reagent Set	25 vials	TNT826

Required apparatus

Description	Quantity	Unit	Catalog number
DRB200 Reactor, 115 V, 9x13 mm + 2x20 mm (mono block)	1	each	DRB20001
OR			
DRB200 Reactor, 230 V, 9x13 mm + 2x20 mm (mono block)	1	each	DRB20005
Pipet, variable volume, 1–5 mL	1	each	BBP065
Pipet Tips, for BBP065 pipet	2	75/pkg	BBP068
Pipet, variable volume, 0.2–1.0 mL	1	each	BBP078
Pipet Tips, for BBP078 pipet	2	100/pkg	BBP079

Recommended standards

Description	Unit	Catalog number
Ammonia Nitrogen Standard Sol., 1000-mg/L NH_3–N	1 L	2354153
Ammonia Nitrogen Standard Sol., 10-mg/L NH_3–N	500 mL	15349
Sodium Hydroxide, 5 N	50 mL	245026
Sulfuric Acid, concentrated	500 mL	97949
Wastewater Mixed Inorganic Standard for NH_3-H, NO_3-N, PO_4, COD, SO_4,TOC	500 mL	2833249
Water, deionized	500 mL	27249
Water, organic-free	500 mL	2641549

Optional reagents and apparatus

Description	Unit	Catalog number
Bottle, sampling, low density poly, w/cap, 500 mL	12/pkg	2087079
DRB200 Reactor, 115 V, 21x13 mm + 4x20 mm (dual block)	each	DRB20002
DRB200 Reactor, 115 V, 12x13 mm + 8x20 mm (dual block)	each	DRB20004
DRB200 Reactor, 230 V, 21x13 mm + 4x20 mm (dual block)	each	DRB20006
DRB200 Reactor, 230 V, 12x13 mm + 8x20 mm (dual block)	each	DRB20008
TNTplus Reactor adapter sleeves, 16-mm to 13-mm diameter	5/pkg	2895805
Test Tube Brush	each	69000
PourRite® Ampule breaker 2-mL	each	2484600
Voluette® Ampule breaker 10 mL	each	2196800
Ammonia Nitrogen Standard Solution, 1-mg/L NH_3–N	500 mL	189149
Ammonia Nitrogen Standard Solution, 100-mg/L NH_3–N	500 mL	2406549
Ammonia Nitrogen Standard Solution, 2-mL PourRite Ampule, 50 mg/L	20/pkg	1479120
Ammonia Nitrogen Standard Solution, 10-mL Voluette Ampules, 10 mg/L	16/pkg	1479110
Ammonia Nitrogen Standard Solution, 10-mL Voluette Ampules, 150 mg/L	16/pkg	2128410

TNTplus Low Range 1 - 16 mg/L N

- Turn on the DRB200 Reactor.
- Set heat to 100°C.

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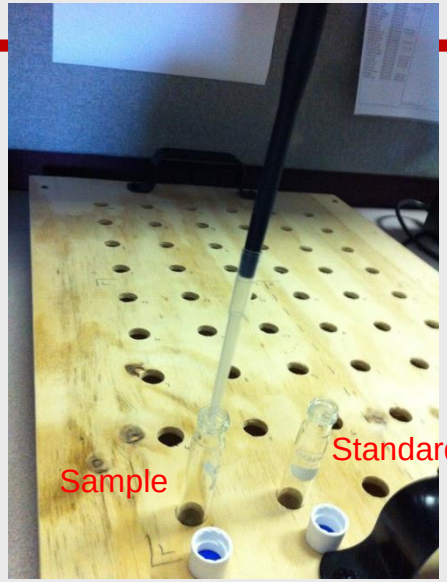
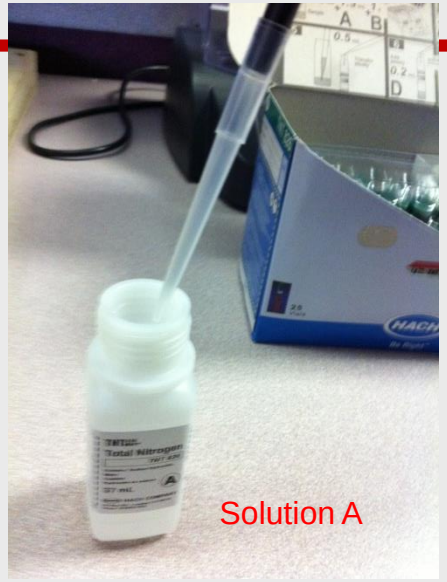
TNTplus Low Range 1 - 16 mg/L N





- Add 1.3 mL of Sample to empty test tube.
- Add 1.3 mL of Solution A to the test tube with the 1.3 mL of Sample.
- Add 1 Reagent B tablet in the test tube and quickly cap vial.

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TNTplus Low Range 1 - 16 mg/L N

- Insert the reaction tubes in the reactor and close the lid.
- Heat 60 minutes for the flat bottom test tubes and for the round bottom test tubes heat for 75 minutes.
- Remove the hot reaction tubes from the reactor and allow the vials to cool to room temperature.

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TNTplus Low Range 1 - 16 mg/L N

- Once the vials are cooled to room temperature, remove the cap and add 1 MicroCap C to the tube.
- Replace the cap and invert 2-3 times until no streaks can be seen in the reaction tube.

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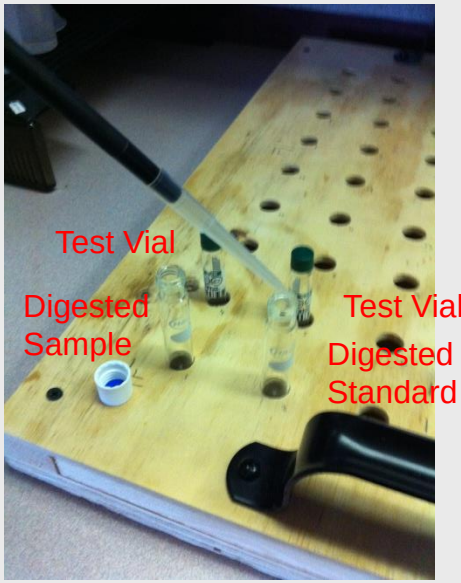
TNTplus Low Range 1 - 16 mg/L N



- Pipet 0.5 mL of the digested sample from the reaction tube into a test vial. Transfer slowly.
- Pipet 0.2 mL of Solution D into the test vial.
- Quickly cap and invert the test vial 2-3 times until no streaks are seen in the vial.

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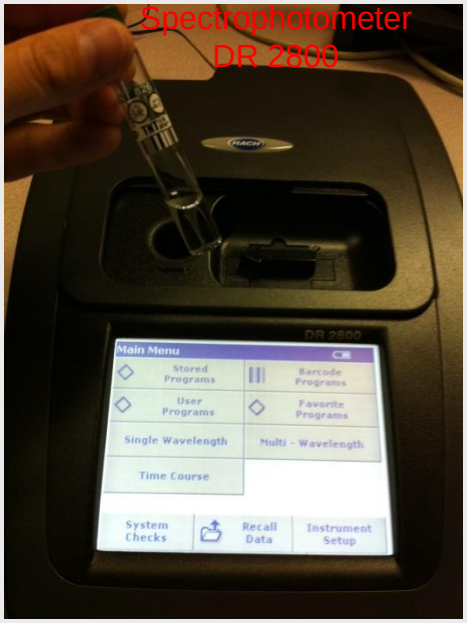
TNTplus Low Range 1 - 16 mg/L N



- Wait 15 minutes after inverting the test vial.
- After 15 minutes wipe the vial and insert the vial into a Spectrophotometer (DR 2800, DR 3800, DR 3900)
- The instrument read the barcode, then selects and performs the correct test. Then results are in mg/L N.

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TNTplus High Range 5 - 40 mg/L N

Nitrogen, Total

DOC316.53.01088

Persulfate Digestion Method — **Method 10208**

HR (5 to 40 mg/L N) — **TNTplus™ 827**

Scope and Application: For water and wastewater

Test preparation

How to use instrument-specific information

The *Instrument-specific information* table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

Table 1 Instrument-specific information

Instrument	Light shield
DR 3900	LZV849
DR 3800, DR 2800	LZV646

Before starting the test:

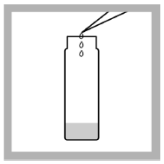
DR 3900, DR 3800, DR 2800: Install the light shield in Cell Compartment #2 before performing this test.
Read the Safety Advice and Expiration Date on the package.
Recommended sample and reagent temperature is 15–25 °C (59–77 °F). Recommended reagent storage temperature is 15–25 °C (59–77 °F).
Recommended sample pH is between 3–12.
Digestion is required for determining total nitrogen.
If the test is not performed at the recommended temperature an incorrect result may be obtained.
Use only high quality deionized water or Organic Free Water for preparing nitrogen standards or making sample dilutions and reagent blanks.
TNTplus methods are activated from the Main Menu when the sample vial is inserted into the sample cell holder.
IMPORTANT: Sodium hydroxide solution A / Oxidant tablet B / MicroCap C: After addition of reagents A, B and C the reagent bottles must be reclosed **immediately**.
IMPORTANT: Reaction Tubes (Ø 20 mm): Do not use reaction tubes more than **13 times**. After use, clean thoroughly with a brush and water, then rinse well with nitrogen-free distilled water and dry.
IMPORTANT: Turbidity: Slight turbidity does not interfere; high turbidity after the addition of the MicroCap C should be allowed to settle before pipetting the digested sample.

Collect the following items:

Description	Quantity
Nitrogen, Total, HR TNT827 Reagent Set	1
DRB200 Reactor, 20-mm wells	1
Light Shield (DR 3900, DR 3800, DR 2800)	1
Pipet for 1–5 mL volumes	1
Pipet Tips for 1–5 mL pipet	1
Pipet for 0.2–1.0 mL volumes	1
Pipet Tips for 0.2–1.0 mL pipet	3

Persulfate digestion method

1. Turn on the DRB200 Reactor and heat to 100 °C.

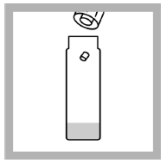


2. Add **0.5 mL** of sample, **2.0 mL** of Solution A and **1** Reagent B tablet in quick succession to a dry 20-mm reaction tube.

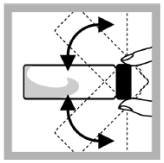
Close the reaction tube immediately. **Do not invert.**

3. Insert the reaction tubes in the reactor and close the lid. Heat for one hour.

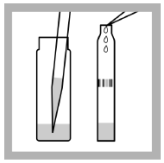
4. Remove the hot reaction tubes from the reactor. Cool the vials to room temperature (15–20 °C).



5. After the reaction tube has cooled, remove the cap and add 1 Micro Cap C to the tube.



6. Cap and invert the reaction tube 2–3 times until no more streaks can be seen in the reaction tube solution.

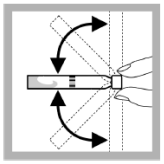


7. Pipet 0.5 mL (500 µL) of the digested sample from the reaction tube into a test vial.

8. Pipet 0.2 mL (200 µL) of Solution D into the test vial.

TNTplus High Range 5 - 40 mg/L N

Persulfate digestion method (continued)



9. Quickly cap and invert the test vial 2–3 times until no more streaks can be seen in the vial solution.

10. Wait 15 minutes.

11. After the timer expires wipe the vial and Insert the prepared vial into the cell holder.

The instrument reads the barcode, then selects and performs the correct test.

Results are in mg/L N.

No instrument zero is required.

Reagent Blanks

A reagent blank can be measured and the value subtracted from the results of each test performed in same reagent lot. Use nitrogen-free deionized water in place of sample in the *Persulfate digestion method* test.

To subtract the value of the blank from a series of measurements:

1. Measure the blank per step 11.
2. Turn on the reagent blank option.
3. The measured value of the blank should be displayed in the highlighted box. Accept this value.

The reagent blank value will now be subtracted from all results until the function is turned off or a different method is selected. Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

Interferences

The ions listed in the *Interfering substances* table have been individually checked up to the given concentrations and do not cause interference. The cumulative effects of these ions or the influence of other ions have not been determined.

Table 2 Interfering substances

Interfering substance	Interference level
COD	1000 mg/L
Chloride	2000 mg/L

Sample collection, preservation and storage

- Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis.
- Preserve the sample by reducing the pH to 2 or less with concentrated (at least 2 mL/L) Sulfuric Acid.
- Store samples at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days.
- Warm stored samples to 15–25 °C and neutralize with 5 N Sodium Hydroxide before analysis.
- Correct the test result for volume additions.

Accuracy check

Standard solution method
Note: *Refer to the instrument user manual for specific software navigation instructions.*

Required for accuracy check:

- 10 mg/L ammonia nitrogen standard

  OR

- Wastewater Influent Mixed Parameters Inorganics Standard

1. Use one of the following in place of the sample.
   - Use 0.5 mL of 10 mg/L standard in place of the sample in step 2.
   - Use 0.5 mL of a Wastewater Influent Mixed Parameters Inorganics Standard in place of the sample in step 2. This standard contains 15 mg/L ammonia nitrogen and 10 mg/L nitrate nitrogen to give a combined standard of 25 mg/L as total nitrogen.
2. Follow the *Persulfate digestion method* test procedure.

Summary of method

Inorganically and organically bonded nitrogen is oxidized to nitrate by digestion with peroxodisulphate. The nitrate ions react with 2,6-dimethylphenol in a solution of sulphuric and phosphoric acid to form a nitrophenol. Test results are measured at 345 nm.

TNTplus High Range 5 - 40 mg/L N

Consumables and replacement items

Required reagents

Description	Unit	Catalog number
Nitrogen Total, HR TNT827 Reagent Set	25 vials	TNT827

Required apparatus

Description	Quantity	Unit	Catalog number
DRB200 Reactor, 115 V, 9x13mm + 2x20 mm (mono block)	1	each	DRB20001
OR			
DRB200 Reactor, 230 V, 9x13mm + 2x20 mm (mono block)	1	each	DRB20005
Pipet, variable volume, 1–5 mL	1	each	BBP065
Pipet Tips, for BBP065 pipet	1	75/pkg	BBP068
Pipet, variable volume, 0.2–1.0 mL	1	each	BBP078
Pipet Tips, for BBP078 Pipet	3	100/pkg	BBP079

Recommended standards

Description	Unit	Catalog number
Ammonia Nitrogen Standard Sol., 1000-mg/L NH_3–N	1 L	2354153
Ammonia Nitrogen Standard Sol., 10-mg/L NH_3–N	500 mL	15349
Sodium Hydroxide, 5 N	50 mL	245026
Sulfuric Acid, concentration	500 mL	97949
Wastewater Mixed Inorganic Standard for NH_3-H, NO_3-N, PO_4, COD, SO_4,TOC	500 mL	2833149
Water, deionized	500 mL	27249
Water, organic-free	500 mL	2641549

Optional reagents and apparatus

Description	Unit	Catalog number
Bottle, sampling, low density poly, w/cap, 500 mL	12/pkg	2087079
DRB200 Reactor, 115 V, 21x13mm + 4x20 mm (dual block)	each	DRB20002
DRB200 Reactor, 115 V, 12x13mm + 8x20 mm (dual block)	each	DRB20004
DRB200 Reactor, 230 V, 21x13mm + 4x20 mm (dual block)	each	DRB20006
DRB200 Reactor, 230 V, 12x13mm + 8x20 mm (dual block)	each	DRB20008
TNTplus Reactor adapter sleeves, 16-mm to 13-mm diameter	5/pkg	2895805
Test Tube Brush	each	69000
PourRite® Ampule breaker 2-mL	each	2484600
Voluette® Ampule breaker 10 mL	each	2196800
Ammonia Nitrogen Standard Solution, 1-mg/L NH_3–N	500 mL	189149
Ammonia Nitrogen Standard Solution, 100-mg/L NH_3–N	500 mL	2406549
Ammonia Nitrogen Standard Solution, 2-mL PourRite Ampule, 50 mg/L	20/pkg	1479120
Ammonia Nitrogen Standard Solution, 10-mL Voluette Ampules, 10 mg/L	16/pkg	1479110
Ammonia Nitrogen Standard Solution, 10-mL Voluette Ampules, 150 mg/L	16/pkg	2128410

TNTplus High Range 5 - 40 mg/L N

- Turn on the DRB200 Reactor.
- Set heat to 100°C.

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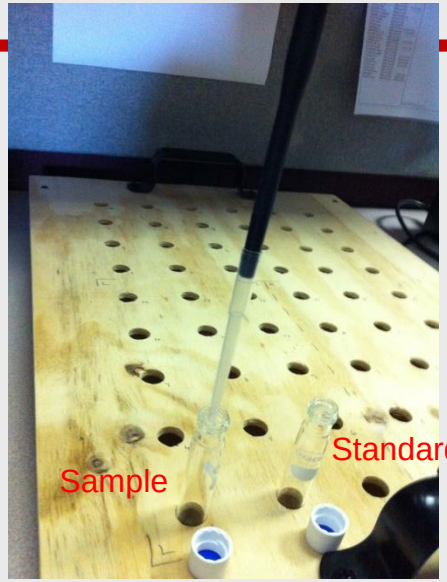
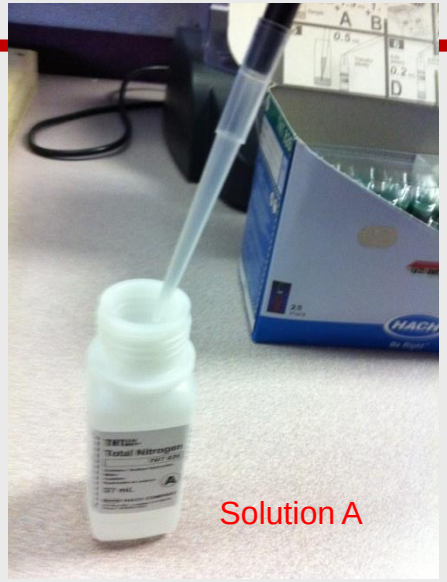
TNTplus High Range 5 - 40 mg/L N





- Add 0.5 mL of Sample to empty test tube.
- Add 2.0 mL of Solution A to the test tube with the 1.3 mL of Sample.
- Add 1 Reagent B tablet in the test tube and quickly cap vial.

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TNTplus High Range 5 - 40 mg/L N

- Insert the reaction tubes in the reactor and close the lid.
- Heat 60 minutes for the flat bottom test tubes and for the round bottom test tubes heat for 75 minutes.
- Remove the hot reaction tubes from the reactor and allow the vials to cool to room temperature.

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TNTplus High Range 5 - 40 mg/L N

- Once the vials are cooled to room temperature, remove the cap and add 1 MicroCap C to the tube.
- Replace the cap and invert 2-3 times until no streaks can be seen in the reaction tube.

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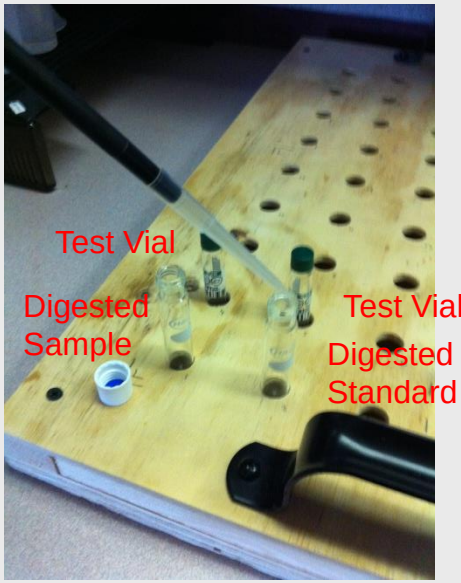
TNTplus High Range 5 - 40 mg/L N



- Pipet 0.5 mL of the digested sample from the reaction tube into a test vial. Transfer slowly.
- Pipet 0.2 mL of Solution D into the test vial.
- Quickly cap and invert the test vial 2-3 times until no streaks are seen in the vial.

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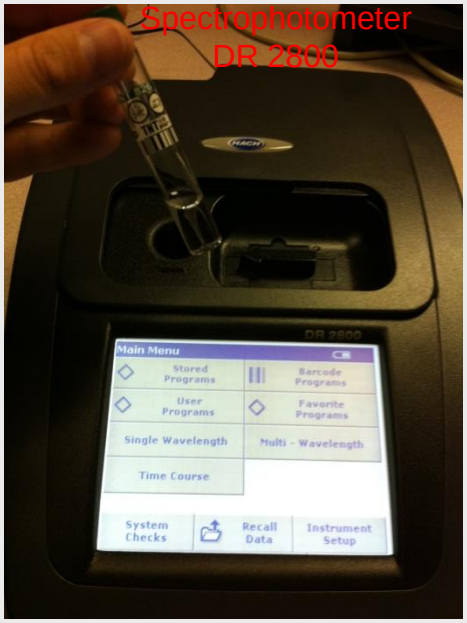
TNTplus High Range 5 - 40 mg/L N



- Wait 15 minutes after inverting the test vial.
- After 15 minutes wipe the vial and insert the vial into a Spectrophotometer (DR 2800, DR 3800, DR 3900)
- The instrument reads the barcode, then selects and performs the correct test. Then results are in mg/L N.

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b. HACH Method 10071 Test ‘N Tubes

Nitrogen, Total

DOC316.53.01086

Persulfate Digestion Method **Method 10071**

0.5 to 25.0 mg/L N (LR) **Test 'N Tube™ Vials**

Scope and application: For water and wastewater.

Test preparation

Instrument-specific information

Table 1 shows all of the instruments that have the program for this test. The table also shows adapter and light shield requirements for the instruments that use them.

To use the table, select an instrument, then read across to find the applicable information for this test.

Table 1 Instrument-specific information for test tubes

Instrument	Adapters	Light shield
DR 6000, DR 5000	—	—
DR 3900	—	LZV849
DR 3800, DR 2800, DR 2700	—	LZV646
DR 1900	9609900 (D[1])	—
DR 900	4846400	Cover supplied with the instrument

[1] The D adapter is not available with all instrument versions.

Before starting

Install the instrument cap on the DR 900 cell holder before ZERO or READ is pushed.
DR 3900, DR 3800, DR 2800 and DR 2700: Install the light shield in Cell Compartment #2 before this test is started.
Digestion is required for total nitrogen determinations.
The vials must be mixed carefully for accurate results. Start each vial inversion with the vial in the vertical position, with the cap on the top. Turn the vial upside-down and wait for all of the solution to flow down to the cap. Return the vial to the vertical position and wait for all of the solution to flow down to the bottom of the vial. This mixing method equals one inversion.
If the test result is over-range, dilute a fresh portion of sample and repeat the complete test procedure. The digestion must be repeated for accurate results.
Use the deionized water that is supplied in the reagent set or organic-free water for the blank vial and for the preparation of standard solutions.
Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.
Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

Items to collect

Description	Quantity
Test 'N Tube LR Total Nigtrogen Reagent Set	1
DRB200 Reactor	1
Finger cots	2

Items to collect (continued)

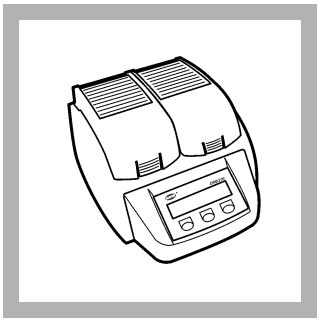
Description	Quantity
Funnel, micro	1
Light shield or adapter (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	1
Pipet, TenSette®, 0.1- to 1.0-mL, with pipet tips	1
Test tube rack	1 to 3

Refer to Consumables and replacement items on page 7 for order information.

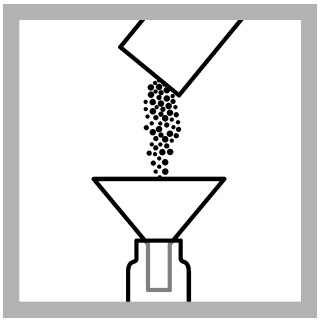
Sample collection and storage

- Collect samples in clean glass or plastic bottles.
- Analyze the samples as soon as possible for best results.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated sulfuric acid (about 2 mL per liter). No acid addition is necessary if the sample is tested immediately.
- Keep the preserved samples at or below 6 °C (43 °F) for a maximum of 28 days.
- Let the sample temperature increase to room temperature before analysis.
- Before analysis, adjust the pH to 7 with 5 N sodium hydroxide solution.
- Correct the test result for the dilution caused by the volume additions.

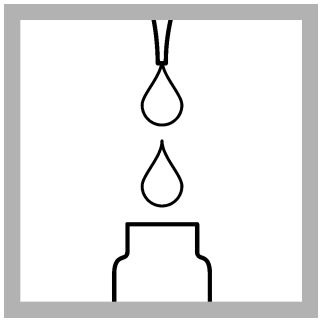
Persulfate digestion for Test 'N Tubes



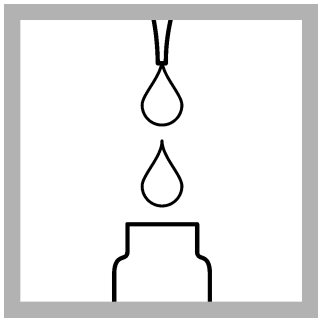
1. Start the DRB200 Reactor. Set the temperature to 105 °C.



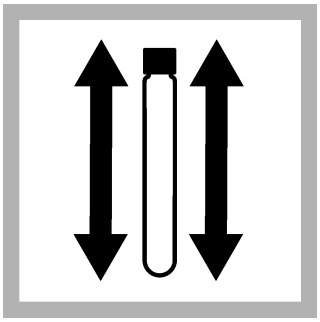
2. Use a funnel to add the contents of one Total Nitrogen Persulfate Reagent Powder Pillow to each of two Total Nitrogen Hydroxide Digestion Reagent vials.
Make sure to clean any reagent that gets on the lip of the vials or on the vial threads.



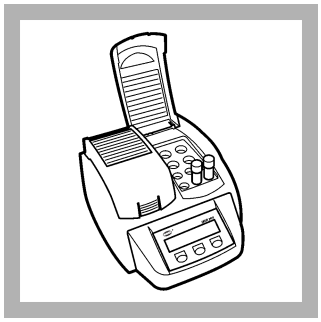
3. Prepare the sample: Add 2 mL of sample to one of the vials.



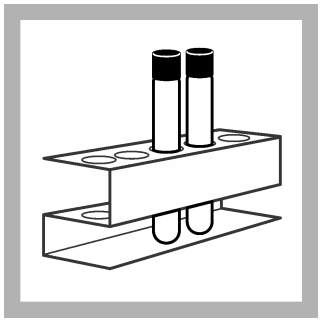
4. Prepare the blank: Add 2 mL of deionized water (included in the kit) to the second vial.
Use only water that is free of all nitrogen-containing species as a substitute for the provided deionized water.



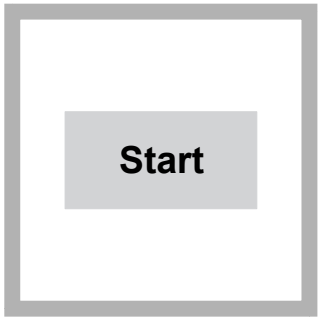
5. Put the caps on both vials. Shake vigorously for at least 30 seconds to mix. Undissolved powder will not affect the accuracy of the test.



6. Put the vials in the reactor and close the lid. Leave the vials in the reactor for exactly 30 minutes.

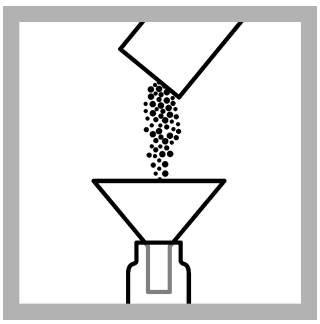


7. At 30 minutes, use finger cots to immediately remove the vials from the reactor. Let the vials cool to room temperature.

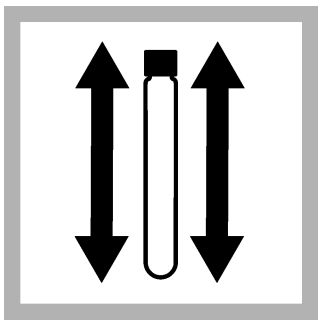


8. Start program **350 N, Total LR TNT**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

Note: *Although the program name can be different between instruments, the program number does not change.*

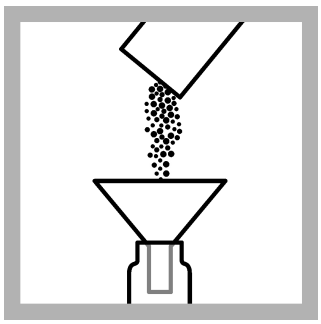


9. Add the contents of one Total Nitrogen (TN) Reagent A Powder Pillow to each vial.

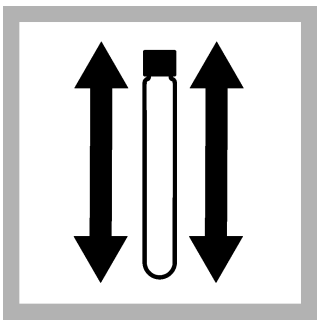


10. Put the caps on both vials. Shake for 15 seconds.

11. Start the instrument timer. A 3-minute reaction time starts.

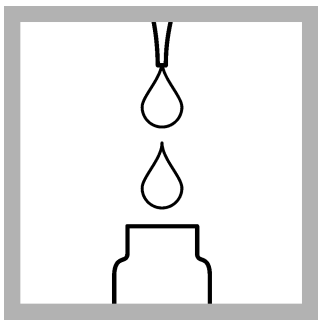


12. After the timer expires, remove the caps from the vials. Add one TN Reagent B Powder Pillow to each vial.

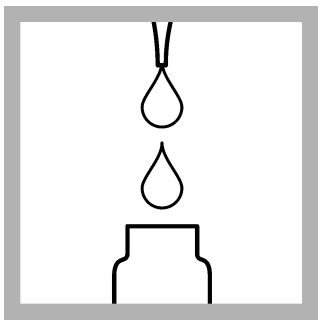


13. Put the caps on both vials. Shake for 15 seconds to mix. The reagent will not dissolve completely. Undissolved powder will not affect the accuracy of the test.
The solution will start to turn yellow.

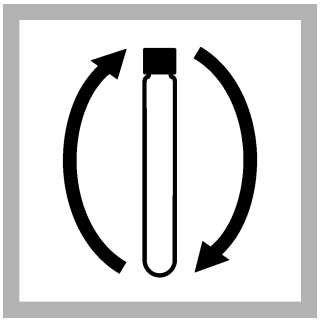
14. Start the instrument timer. A 2-minute reaction time starts.



15. Prepared sample: When the timer expires, use a pipet to put 2 mL of the digested, treated prepared **sample** into one TN Reagent C vial.

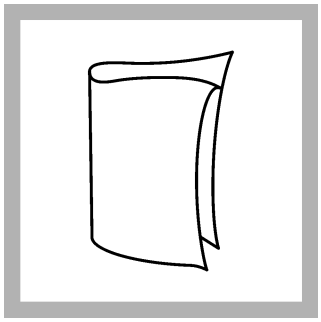


16. Blank: When the timer expires, use a pipet to put 2 mL of the digested, treated **blank** into the second TN Reagent C vial.

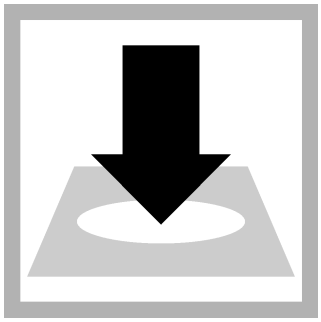


17. Put the caps on both vials. Invert 10 times to mix. Use slow, deliberation inversions for complete recovery. The vials will be warm to the touch.

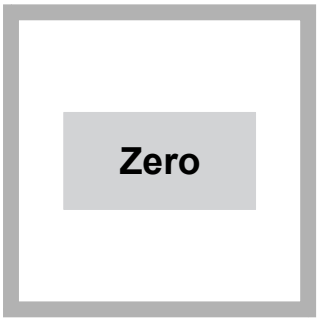
18. Start the instrument timer. A 5-minute reaction time starts.
The yellow color will intensify.



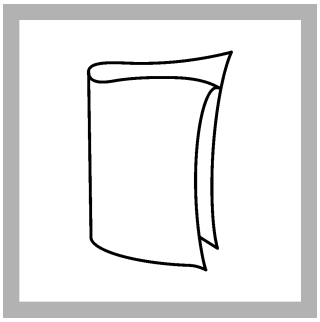
19. When the timer expires, clean the blank vial.



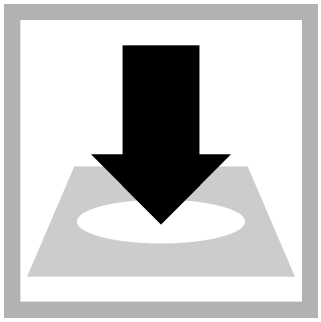
20. Insert the blank vial into the 16-mm cell holder.



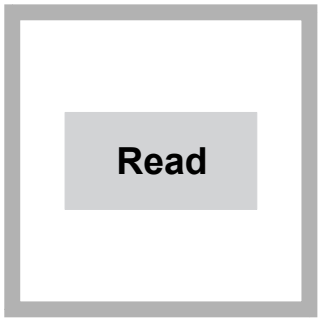
21. Push **ZERO**. The display shows 0.0 mg/L N. Multiple samples can be measured after "zero" is set with the blank.



22. Clean the sample vial.



23. Insert the sample vial into the 16-mm cell holder.



24. Push **READ**. Results show in mg/L N.

Blanks for colorimetric measurement

The reagent blank can be used for up to 7 days for measurements that use the same lot of reagents. Keep the reagent blank in the dark at room temperature (18–25 °C). If a small amount of white floc appears within a week, discard the reagent blank and prepare a new one.

Interferences

The substances in the Table 2 have been tested and found not to interfere up to the indicated levels (in mg/L). Interfering substances that resulted in a concentration change of ±10% appear in the Table 3.

Table 2 Non-interfering substances

Interfering substance	Interference level
Barium	2.6 mg/L
Calcium	300 mg/L
Chromium ($^{3+}$)	0.5 mg/L
Iron	2 mg/L
Lead	6.6 µg/L
Magnesium	500 mg/L
Organic Carbon	150 mg/L
Phosphorus	100 mg/L

Table 2 Non-interfering substances (continued)

Interfering substance	Interference level
Silica	150 mg/L
Silver	0.9 mg/L
Tin	1.5 mg/L

Table 3 Interfering substances

Interfering substance	Interference level
Bromide	> 60 mg/L; positive interference
Chloride	> 1000 mg/L; positive interference

This test performed with standard nitrogen solutions prepared from the following compounds obtained 95% recovery:

- Ammonium chloride
- Ammonium sulfate
- Ammonium acetate
- Glycine
- Urea

Ammonium chloride or nicotinic-PTSA spikes in domestic influent, effluent and the ASTM standard specification for substitute wastewater (D 5905-96) also resulted in ≥ 95% recovery.

The large amounts of nitrogen-free organic compounds in some samples may decrease digestion efficiency by consuming some of the persulfate reagent. Samples known to contain high levels of organics should be diluted and re-run to verify digestion efficiency.

Accuracy check

Digestion method

For proof of accuracy use Primary Standards for Kjeldahl Nitrogen. This method generally gives 95–100% recovery on organic nitrogen standards. Analysts have found Nicotinic acid-PTSA (p-Toluenesulfonate) to be the most difficult to digest. Other compounds may yield different percent recoveries.
Items to collect:

- Primary Standard for Kjeldahl Nitrogen (Ammonia-PTSA, Glycine-PTSA or Nicotinic-PTSA)
- 1-L volumetric flask, Class A
- Deionized water (use the deionized water supplied in the reagent set or water that is free of all organic and nitrogen-containing species)

1. Prepare a 25-mg/L N equivalent standard.
   a. Weigh the applicable standard:
      - Ammonia-PTSA: 0.3379 g
      - Glycine-PTSA: 0.4416 g
      - Nicotinic-PTSA: 0.5274 g
   b. Use a funnel to add the standard to the volumetric flask.
   c. Add deionized water to the flask and mix to dissolve the standard.
   d. Dilute to the mark with deionized water. Mix well.
2. Use the test procedure to measure the concentration of the nitrogen standard. Calculate the percent recovery as follows:

   % recovery = [(measured concentration)/25] x 100

   Note: *The minimum expected % recovery for each standard is 95%.*

Standard additions method (sample spike)

Use the standard additions method (for applicable instruments) to validate the test procedure, reagents and instrument and to find if there is an interference in the sample.

Items to collect:

- Ammonia Nitrogen Standard Solution, 1000-mg/L as NH_3–N
- Ampule breaker
- Pipet, TenSette®, 0.1–1.0 mL and tips
- 50-mL mixing cylinders (3)

1. Use the test procedure to measure the concentration of the sample, then keep the (unspiked) sample in the instrument.
2. Go to the Standard Additions option in the instrument menu.
3. Select the values for standard concentration, sample volume and spike volumes.
4. Open the standard solution.
5. Prepare three spiked samples: use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3 mL of the standard solution, respectively, to three 50-mL portions of fresh sample. Mix well.
6. Use the test procedure to measure the concentration of each of the spiked samples. Start with the smallest sample spike. Measure each of the spiked samples in the instrument.
7. Select **Graph** to compare the expected results to the actual results.

   Note: *If the actual results are significantly different from the expected results, make sure that the sample volumes and sample spikes are measured accurately. The sample volumes and sample spikes that are used should agree with the selections in the standard additions menu. If the results are not within acceptable limits, the sample may contain an interference.*

Standard solution method

Use the standard solution method to validate the test procedure, the reagents and the instrument.

Items to collect:

- 10-mg/L ammonia nitrogen standard solution

1. Use the test procedure to measure the concentration of the standard solution.
2. Compare the expected result to the actual result.

   Note: *The factory calibration can be adjusted slightly with the standard adjust option so that the instrument shows the expected value of the standard solution. The adjusted calibration is then used for all test results. This adjustment can increase the test accuracy when there are slight variations in the reagents or instruments.*

Method performance

The method performance data that follows was derived from laboratory tests that were measured on a spectrophotometer during ideal test conditions. Users can get different results under different test conditions.

Program	Standard	Precision (95% confidence interval)	Sensitivity Concentration change per 0.010 Abs change
350	10 mg/L NH_3–N	9.6–10.4 mg/L N	0.5 mg/L N

Summary of method

An alkaline persulfate digestion converts all forms of nitrogen to nitrate. Sodium metabisulfite is added after the digestion to eliminate halogen oxide interferences. Nitrate then reacts with chromotropic acid under strongly acidic conditions to form a yellow complex. The measurement wavelength is 410 nm for spectrophotometers or 420 nm for colorimeters.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
Nitrogen, Total, LR, Test 'N Tube™ Reagent Set		50 vials	2672245

Required apparatus

Description	Quantity/test	Unit	Item no.
DRB 200 Reactor, 110 VAC option, 15 x 16-mm wells	1	each	LTV082.53.40001
OR			
DRB 200 Reactor, 220 VAC option, 15 x 16-mm wells	1	each	LTV082.52.40001
Funnel, micro, poly	1	each	2584335
Pipet, TenSette®, 0.1–1.0 mL	1	each	1970001
Pipet tips, for TenSette® Pipet, 0.1–1.0 mL	2	50/pkg	2185696
Test tube rack	1	each	1864100
Finger cots	2	2/pkg	1464702

Recommended standards

Description	Unit	Item no.
Nitrogen, Ammonia Standard Solution, 1000-mg/L NH_3-N	1 L	2354153
Nitrogen Ammonia Standard Solution, 10-mg/L NH_3–N	500 mL	15349
Kjeldahl Nitrogen Primary Standard Set	set of 3	2277800
Wastewater Influent Standard Solution, Mixed Parameter, for NH_3-N, NO_3-N, PO_4, COD, SO_4, TOC	500 mL	2833149
Water, deionized	500 mL	27249
Water, organic-free	500 mL	2641549

Optional reagents and apparatus

Description	Unit	Item no.
Balance, analytical, 80 g x 0.1 mg 100–240 VAC	each	2936701
Mixing cylinder, graduated, 50-mL	each	2088641
Flask, volumetric, Class A, 1000-mL glass	each	1457453
Sodium Hydroxide Solution, 5 N	50 mL	245026
Sulfuric Acid, ACS	500 mL	97949
Ampule Breaker, 2-mL PourRite® Ampules	each	2484600
Ampule Breaker, 10-mL Voluette® Ampules	each	2196800
Nitrogen Ammonia Standard Solution, 1.0-mg/L NH_3–N	500 mL	189149
Nitrogen, Ammonia Standard Solution, 100-mg/L NH_3-N	500 mL	2406549
Nitrogen, Ammonia Standard Solution, 2-mL PourRite Ampule, 50 mg/L	20/pkg	1479120
Nitrogen, Ammonia Standard Solution, 10-mL Voluette Ampules, 50 mg/L	16/pkg	1479110
Nitrogen, Ammonia Standard Solution, 10-mL Voluette® Ampules, 150 mg/L	16/pkg	2128410
Pipet, TenSette®, 1.0–10.0 mL	each	1970010

Optional reagents and apparatus (continued)

Description	Unit	Item no.
Pipet tips for TenSette® Pipet, 1.0–10.0 mL	50/pkg	2199796
Pipet tips for TenSette® Pipet, 1.0–10.0 mL	250/pkg	2199725
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	1000/pkg	2185628

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10/2014, Edition 10

c. HACH Method TNT 826

Nitrogen, Total

DOC316.53.01087

Persulfate Digestion Method

Method 10208

1 to 16 mg/L N (LR)

TNTplus™ 826

Scope and application: For water and wastewater.

Test preparation

Instrument-specific information

Table 1 shows all of the instruments that have the program for this test. The table also shows the adapter and light shield requirements for the applicable instruments that can use TNTplus vials.

To use the table, select an instrument, then read across to find the applicable information for this test.

Table 1 Instrument-specific information for TNTplus vials

Instrument	Adapters	Light shield
DR 6000, DR 5000	—	—
DR 3900	—	LZV849
DR 3800, DR 2800	—	LZV646
DR 1900	9609900 or 9609800 (A)	—

Before starting

DR 3900, DR 3800, DR 2800: Install the light shield in Cell Compartment #2 before this test is started.
Review the safety information and the expiration date on the package.
The recommended sample pH is 3–12.
The sample temperature must be 15–25 °C (59–77 °F) for accurate results.
The recommended temperature for reagent storage is 15–25 °C (59–77 °F).
Important: Make sure to close reagent bottles A, B and C immediately after each use.
The 20-mm reaction tube can be used a maximum of 13 times. After each use, clean the tube thoroughly with a brush and water, then rinse well with high-quality distilled water and let dry.
If a large amount of turbidity forms after the addition of MicroCap C, let the turbidity settle, then go to the next step. A small amount of turbidity does not interfere.
Use only high-quality deionized water or organic-free water for standards preparation, sample dilutions or reagent blanks.
DR 1900: Go to All Programs>LCK or TNTplus Methods>Options to select the TNTplus number for the test. Other instruments automatically select the method from the barcode on the vial.
Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.
Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

Items to collect

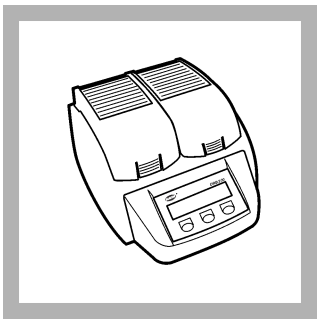
Description	Quantity
Nitrogen, Total, LR TNTplus Reagent Set	1
DRB200 reactor with 20-mm wells	1
Pipet, adjustable volume, 1.0–5.0 mL	1
Pipet, adjustable volume, 0.2–1.0 mL	1
Pipet tips	1
Test tube rack	1

Refer to Consumables and replacement items on page 4 for order information.

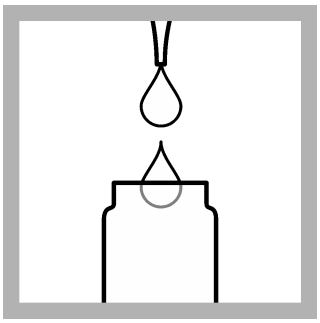
Sample collection and storage

- Collect samples in clean glass or plastic bottles.
- Analyze the samples as soon as possible for best results.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated sulfuric acid (approximately 2 mL per liter). No acid addition is necessary if the sample is tested immediately.
- Keep the preserved samples at or below 6 °C (43 °F) for a maximum of 28 days.
- Let the sample temperature increase to room temperature before analysis.
- Before analysis, adjust the pH to 7 with 5 N sodium hydroxide solution.
- Correct the test result for the dilution caused by the volume additions.

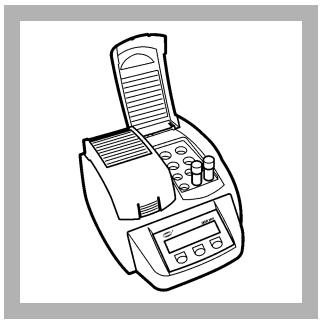
Test procedure



1. Set the DRB200 reactor power to on. Set the temperature to 100 °C.

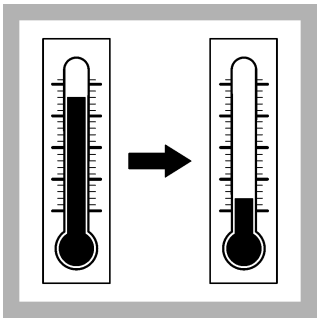


2. Add 1.3 mL of sample, 1.3 mL of Solution A and 1 Reagent B tablet in quick succession to a dry 20-mm reaction tube.
Close the reaction tube immediately. Do not invert.

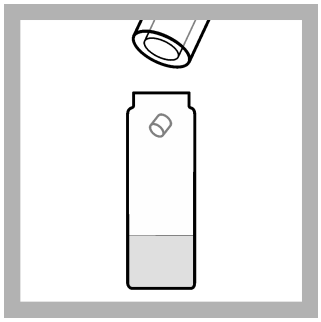


3. Insert the reaction tube in the preheated DRB200 reactor. Close the lid.

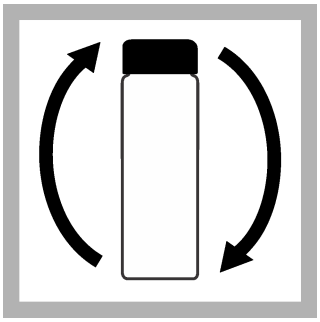
4. Keep the reaction tube in the reactor for 1 hour.



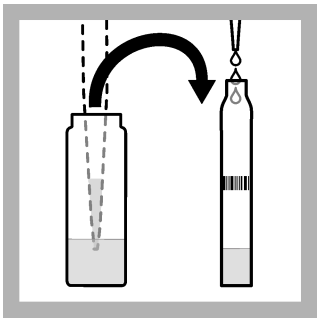
5. When the timer expires, carefully remove the reaction tube from the reactor. Let the temperature of the reaction tube decrease to room temperature.



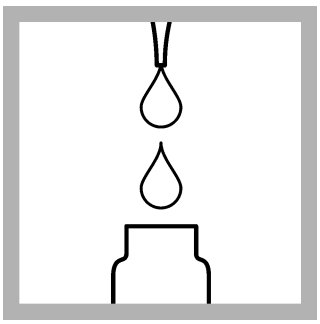
6. When cool, add 1 Micro Cap C to the reaction tube.



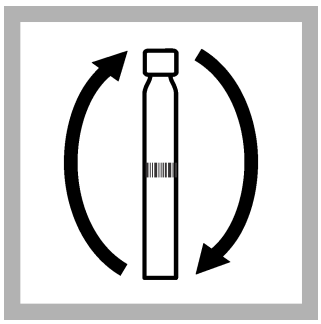
7. Tighten the cap on the reaction tube and invert until completely mixed.



8. Use a pipet to add 0.5 mL of the digested sample from the 20-mm reaction tube into a test vial.

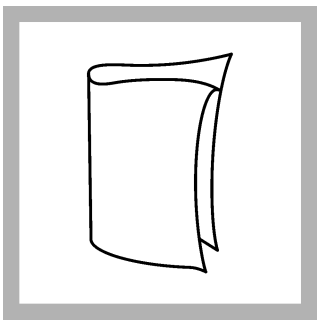


9. Use a pipet to add 0.2 mL of Solution D to the test vial.

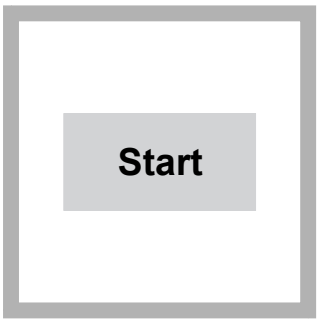


10. Quickly tighten the cap on the vial and invert until completely mixed.

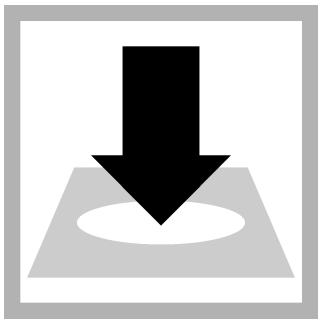
11. Start the reaction time of 15 minutes.



12. When the timer expires, clean the vial.



13. DR 1900 only: Select program 826. Refer to Before starting on page 1.



14. Insert the vial into the cell holder. DR 1900 only: Push **READ**.
Results show in mg/L N.

Reagent blank correction

For the best results, measure the reagent blank value for each new lot of reagent. Replace the sample with deionized water in the test procedure to determine the reagent blank value. Subtract the reagent blank value from the sample results automatically with the reagent blank adjust option. Measure the reagent blank value when a new lot of reagent is used.

1. Use deionized water as the sample in the test procedure to measure the reagent blank value.
2. Set the reagent blank function to on. The measured reagent blank value is shown.

3. Accept the blank value. The reagent blank value is then subtracted from all results until the reagent blank function is set to off or a different method is selected.
   Note: *As an alternative, record or enter the reagent blank value at a different time. Push the highlighted reagent blank box and use the keypad to enter the value.*

Interferences

Table 2 shows that the ions were individually examined to the given concentrations and do not cause interference. No cumulative effects or influences of other ions were found.

Table 2 Interfering substances

Interfering substance	Interference level
COD	400 mg/L
Chloride	800 mg/L

Accuracy check

Standard solution method

Use the standard solution method to validate the test procedure, the reagents and the instrument.

Items to collect:

- Nitrogen, Ammonia Standard Solution, 10-mg/L NH_3–N or Wastewater Effluent Standard Solution, Mixed Parameter (contains 2-mg/L NH_3–N and 4-mg/L NO_3^-–N to give a combined 6-mg/L total nitrogen)

1. Use the test procedure to measure the concentration of the standard solution.
2. Compare the expected result to the actual result.
   Note: *The factory calibration can be adjusted slightly with the standard adjust option so that the instrument shows the expected value of the standard solution. The adjusted calibration is then used for all test results. This adjustment can increase the test accuracy when there are slight variations in the reagents or instruments.*

Summary of Method

Inorganic and organic nitrogen compounds are digested with peroxodisulfate and are oxidized to nitrate. The nitrate ions react with 2,6-dimethylphenol in a solution of sulfuric and phosphoric acid to form a nitrophenol. The measurement wavelength is 345 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
Nitrogen, Total, LR TNTplus Reagent Set	1	25/pkg	TNT826

Required apparatus

Description	Quantity/test	Unit	Item no.
DRB 200 Reactor, 115 VAC option, 9 x 13 mm + 2 x 20 mm, 1 block	1	each	DRB20001
DRB 200 Reactor, 230 VAC option, 9 x 13 mm + 2 x 20 mm, 1 block	1	each	DRB20005
Pipet, adjustable volume, 1.0–5.0 mL	1	each	BBP065
Pipet tips, for 1.0–5.0 mL pipet	1	75/pkg	BBP068
Pipet, adjustable volume, 0.2–1.0 mL	1	each	BBP078
Pipet tips, for 0.2–1.0 mL pipet	2	100/pkg	BBP079
Test tube rack	1	each	1864100

Required apparatus (continued)

Description	Quantity/test	Unit	Item no.
Light shield, DR 3800, DR 2800, DR 2700	1	each	LZV646
Light shield, DR 3900	1	each	LZV849

Recommended standards

Description	Unit	Item no.
Nitrogen Ammonia Standard Solution, 10-mg/L NH_3–N	500 mL	15349
Wastewater Effluent Standard Solution, Mixed Parameter, for NH_3-N, NO_3-N, PO_4^{3-}, COD, SO_4^{2-}, TOC	500 mL	2833249

Optional reagents and apparatus

Description	Unit	Item no.
Brush, test tube	each	69000
Sampling bottle with cap, low density polyethylene, 500-mL	12/pkg	2087079
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	245032
Sulfuric Acid, concentrated, ACS	500 mL	97949
Water, deionized	4 L	27256
Water, organic-free	500 mL	2641549

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02/2014, Edition 8

6. NITRATE – N

a. Testing Procedures

Testing Methods – Nitrate Nitrogen

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Testing Procedures

Nitrate Nitrogen

TNTplus Low Range 0.23 – 13.50 mg/L NO_3-N, Method 10206 (TNT 835)

TNTplus Low Range 0.23 – 13.50 mg/L NO_3-N, Method 10206

Nitrate

DOC316.53.01070

Dimethylphenol Method **Method 10206**

LR (0.23 to 13.50 mg/L NO_3–N or 1.00 to 60.00 mg/L NO_3)

TNTplus 835

Scope and Application: For wastewater, drinking water, surface water and process water

Test preparation

How to use instrument-specific information

The *Instrument-specific information* table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

Table 1 Instrument-specific information

Instrument	Light shield
DR 3900	LZV849
DR 3800, DR 2800	LZV646

Before starting the test:

Install the light shield if applicable (see *Instrument-specific information*).
Always read the Safety Advice and Expiration Date on package.
Perform this test at the recommended temperature to avoid an incorrect result. Recommended sample and reagent temperature is 20–23 °C (68–73.4 °F). Analyze samples as soon as possible.
Recommended sample pH is 3–10.
Recommended reagent storage is 15–25 °C (59–77 °F).
TNTplus methods are activated from the Main Menu when the sample vial is inserted into the sample cell holder.

Collect the following items:

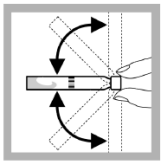
Description	Quantity
Light Shield (see *Instrument-specific information*)	1
Nitrate LR TNT 835 Reagent Set	1
Pipet, variable, 0.2–1.0 mL	1
Pipet Tip, for 0.2–1.0 mL pipet	2

See *Consumables and replacement items* for reorder information.

Dimethylphenol method

1. Pipet 1.0 mL of sample into the reagent vial.

2. Pipet 0.2 mL of Solution A into the vial.



3. Cap and invert the reaction tube 2–3 times until no more streaks can be seen in the reaction tube solution.

4. Wait 15 minutes.

5. After the timer expires wipe the vial and insert the prepared vial into the cell holder. The instrument reads the barcode, then selects and performs the correct test.

No Zero is required. Results are in mg/L NO_3–N

Interferences

The items listed in the *Interfering substances* table have been individually checked up to the given concentrations and do not cause interference. The cumulative effects and influence of other ions have not been determined. High loads of oxidizable organic substances (COD) cause the reagent to change color and to give high-bias results. The test can thus only be used for wastewater analyses if the COD is less than 500 mg/L. Measurement results can be verified using sample dilutions or standard additions.

Nitrite concentrations of more than 2.0 mg/L interfere (high-bias results). Add 50 mg of sulfamic acid (amidosulfonic acid) to 5.0 mL of sample, dissolve and wait for 10 minutes. Analyze the prepared sample as described in the procedure above.

TNTplus Low Range 0.23 – 13.50 mg/L NO_3-N, Method 10206

Table 2 Interfering substances

Interfering substance	Interference level	Interfering substance	Interference level
Ag^{+}	100 mg/L	Cu^{2+}	50 mg/L
Cl^{-}	500 mg/L	Ca^{2+}	50 mg/L
Fe^{3+}	50 mg/L	NO_2^{-}	2 mg/L
K^{+}	500 mg/L	Cd^{2+}	50 mg/L
Na^{+}	500 mg/L	Sn^{2+}	50 mg/L
Ni^{2+}	50 mg/L	Cr^{6+}	5 mg/L
Pb^{2+}	50 mg/L	Fe^{2+}	10 mg/L
Zn^{2+}	50 mg/L	Co^{2+}	10 mg/L

Reagent blanks

A reagent blank can be measured and the value subtracted from the results of each test performed using the same reagent lot number. Use deionized water in place of sample and run the *Dimethylphenol method* procedure as described.

To subtract the value of the blank from a series of measurements:

1. Measure the blank per step 5.
2. Turn on the reagent blank function. The measured value of the blank should be displayed in the highlighted box.
3. Accept the blank value. The reagent blank value will be subtracted from all results until the function is turned off or a different method is selected.

Alternately, the blank can be recorded and entered at any later time by pressing the highlighted reagent blank box and using the keypad to enter the value.

Sample blanks

Colored or turbid samples can cause high results. To compensate for color or turbidity, the procedure is repeated and the color forming reagent that is present in Solution A is not added.

To determine the sample blank:

1. Perform the *Dimethylphenol method* with 0.2 mL of deionized water in place of the 0.2 mL of Solution A in step 2. Use the original cap to cap the sample vial.
2. Subtract the value obtained in step 5 from the value obtained on the original sample to give the corrected sample concentration.

Alternatively, samples that contain only turbidity may be first filtered through a membrane filter and then analyzed.

Samples without color or turbidity do not require sample blanks.

Sample collection, preservation and storage

- Collect samples in clean plastic or glass bottles.
- Analyze samples as soon as possible to prevent bacterial degradation of the nitrate. If immediate analysis is not possible, store at 4 °C (39 °F) or lower if the sample is to be analyzed within 24 to 48 hours. For longer storage periods (up to 14 days), adjust sample pH to 2 or less with Sulfuric Acid, ACS* (about 2 mL per liter). Sample refrigeration is still required.
- Before testing the stored sample, warm to 20–23 °C and neutralize with 5.0 N Sodium Hydroxide Standard Solution*. Do not use mercury compounds as preservatives.
- Correct the test result for volume additions.

Accuracy check

Standard solution method

Note: *Refer to the instrument user manual for specific software navigation instructions.*

Required for accuracy check:

- Nitrate Nitrogen Standard, 10 mg/L, NO_3–N

or

- Wastewater Influent Mixed Parameters Inorganics Standard

1. Use 1.0 mL of Nitrate nitrogen standard, 10 mg/L in place of the sample in step 1.

or

1. Use 1.0 mL of Wastewater Influent Mixed Parameters Inorganics Standard in place of the sample in step 1. This standard contains 10 mg/L nitrate nitrogen combined with ammonia, phosphate, sulfate and organic material.
2. Follow the *Dimethylphenol method* test procedure.

Summary of method

Nitrate ions in solutions containing sulfuric and phosphoric acids react with 2,6-dimethylphenol to form 4-nitro-2,6-dimethylphenol. Test results are measured at the wavelengths in the *Test wavelengths* table.

Table 3 Test wavelengths

Instrument	Wavelength
DR 5000	370 nm
DR 3900, DR 3800, DR 2800	345 nm

* See *Optional reagents and apparatus*.

TNTplus Low Range 0.23 – 13.50 mg/L NO_3-N, Method 10206

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
Nitrate TNTplus, LR TNT 835	1	25/pkg	TNT835

Required apparatus

Description	Quantity	Unit	Catalog number
Pipet, variable volume, 0.2–1.0 mL	1	each	BBP078
Pipet Tips, for BBP078 pipet	2	100/pkg	BBP079

Recommended standards

Description	Unit	Catalog number
Nitrate Nitrogen Standard Solution, 10-mg/L	500 mL	30749
Nitrate Nitrogen Standard Solution, 1000 mg/L	500 mL	1279249
Wastewater Influent Inorganics Standard for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	2833149
Water, deionized	4 L	27256

Optional reagents and apparatus

Description	Unit	Catalog number
Balance, AccuLab VI–Series, 120 g capacity	each	2694700
Bottle, sampling, low density poly, w/cap, 500 mL, 12/pkg	12/pkg	2087079
Filter Holder, glass, for vacuum filtration	each	234000
Filter, membrane, 47 mm; 0.45-micron	each	2894700
Flask, filtering, glass	1000 mL	54653
Sodium Hydroxide, 5.0 N	50 mL SCDB	245026
Sulfamic Acid	454 g	234401
Sulfuric Acid ACS, concentrated	500 mL	97949
Test Tube Rack for 13-mm vials	each	2497900
Tubing, rubber	12-ft	56019
Aspirator	each	213100

TNTplus Low Range 0.23 – 13.50 mg/L NO_3-N, Method 10206

- Pipet 1.0 mL of sample int the reagent vial.
- Pipet 0.2 mL of Solution A into the vial.
- Cap and invert the reaction tube 2-3 time until no streaks are seen.
- Wait 15 minutes.

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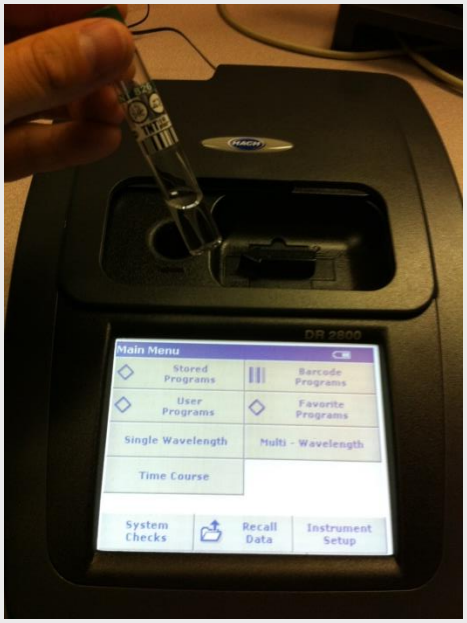
TNTplus Low Range 0.23 – 13.50 mg/L NO_3-N, Method 10206



- After 15 minutes wipe the vial and insert the vial into a Spectrophotometer (DR 2800, DR 3800, DR 3900)
- The instrument reads the barcode, then selects and performs the correct test. Then results are in mg/L N.

b. HACH Method TNT 835

Hach Company TNTplus 835/836 Nitrate Method 10206

Spectrophotometric Measurement of Nitrate in Water and Wastewater

Hach Company TNTplus 835/836 Method 10206

Revision 2.2
January 15, 2013

Spectrophotometric Measurement of Nitrate in Water and Wastewater

1.0 Scope and Application

1.1 These procedures cover the determination of nitrate in drinking water, surface water, domestic and industrial wastes.

1.2 The method is applicable in the range from 0.20 to 35.0 mg/L NO_3^- N.

1.3 This method is equivalent or better in performance to SM 4500-NO_3^- E, EPA 353.2, and EPA 300.0 for the purposes of regulatory reporting of nitrate and nitrate-nitrite.

2.0 Summary of Method

2.1 The Hach TNTplus Nitrate chemistry follows classical electrophillic aromatic substitution in that nitrate in the presence of sulfuric acid yields a nitronium ion ($^+NO_2$) and HSO_4^-. Nitronium ions are electrophiles that attack the aromatic ring of the dimethylphenol reagent to form intermediate nitro-carbonium ions. The basic HSO_4^- ion then extracts a hydrogen ion from the nitro-carbonium intermediate to yield a stable substitution product (o, or p-nitro-dimethylphenol). The nitrodimethylphenol product is a highly colored (directly related to the nitro functional group), quantifiable by its visible absorption spectra. Test results are measured at 345 nm.

3.0 Interferences

3.1 The items listed in the *Interfering substances* table have been individually checked up to the given concentrations and do not cause interference. The cumulative effects and influence of other ions have not been determined. High loads of oxidizable organic substances cause the reagent to change color and to give high-bias results. The test can thus only be used for wastewater analyses if the chemical oxygen demand (COD) is less than 500 mg/L. Measurement results can be verified using sample dilutions or standard additions.

3.2 Nitrite concentrations of more than 2.0 mg/L interfere (high-bias results). Add 50 mg of sulfamic acid (amidosulfonic acid) to 5.0 mL of sample, dissolve and wait for 10 minutes. Analyze the prepared sample as described in the procedure above.

Interfering substance	Interference level (mg/L)	Interfering substance	Interference level (mg/L)
Ag^+	100	Cu^{2+}	50
Cl^-	500	Ca^{2+}	50
Fe^{3+}	50	NO_2^-	2
K^+	500	Cd^{2+}	50
Na^+	500	Sn^{2+}	50
Ni^{2+}	50	Cr^{6+}	5
Pb^{2+}	50	Fe^{2+}	10
Zn^{2+}	50	Co^{2+}	10

3.3 Residual chlorine does not cause an interference with this method.

4.0 Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest possible level. It is suggested that the laboratory perform personal hygiene monitoring of each analyst using this method and that the results of this monitoring be made available to the analyst.

4.2 Unknown samples may contain high concentrations of volatile toxic compounds. Sample containers should be opened in a hood and handled with gloves to prevent exposure.

4.3 This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of any chemicals specified in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses. Additional information on laboratory safety can be found in Sections 16.3 and 16.4.

5.0 Equipment

Note: *Brand names, suppliers, and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using apparatus and materials other than those specified here, but demonstration of equivalent performance that meets the requirements of this method is the responsibility of the laboratory.*

5.1 Sampling equipment

5.1.1 Sample collection bottles – Preferably use polyethylene bottles for collecting and storing samples for nitrate analysis. Glass bottles are satisfactory if previously they have not contained high-nitrate solutions.

5.1.2 Cleaning

5.1.2.1 All glassware used should be washed with hot 1:1 HCl and rinsed with distilled water. Preferably, this glassware should be used only for the determination of nitrate and after use it should be rinsed with distilled water and kept covered until needed again. If this is done, the treatment with 1:1 HCl is only occasionally required.

6.0 Equipment for sample analysis

6.1 Hach Company DR 5000, DR 3800, or DR 2800 spectrophotometer

6.2 Equipment for standard preparation

6.2.1 Volumetric flask – Glass, 1000-mL.

6.2.2 Volumetric pipette – Glass, assorted sizes.

7.0 Reagents and Standards

7.1 Reagent water – Water in which nitrate is not detected at or above the method level of this method. Bottled distilled water, or water prepared by passage of tap water through ion exchange and activated carbon have been shown to be acceptable sources of reagent water.

7.2 Hach Company TNTplus Nitrate Reagent, Cat. No. TNT835 or TNT836.

7.3 Hach Company Nitrate Standard Solutions: 100 mg/L as NO_3^--N (Cat. No. 194749) and 1000 mg/L as NO_3^--N (Cat. No.1279249).

7.4 Method detection limit (MDL) solution

7.4.1 Prepare 7 or more replicate MDL solutions by diluting 3.0 mL of the 100 mg/L standard spiking solution (Section 7.3) to 1000 mL. Final concentration = 0.3 mg NO_3^--N /L.

7.5 Initial precision and recovery (IPR) solution

7.5.1 Prepare 4 or more replicate IPR solutions by diluting 5.0 mL of the 1000 mg/L standard spiking solution (Section 7.3) to 1000 mL. Final concentration = 5 mg NO_3^--N /L.

8.0 Sample Collection, Preservation and Storage

8.1 Samples may be collected in clean glass or plastic bottles.

8.2 Analyze samples as soon as possible. If immediate analysis is not possible, store at 4º C or cooler and analyze within 48 hours.

8.3 If longer storage is required (up to 14 days), adjust sample pH to less than 2 with sulfuric acid (about 2 mL per liter). Sample refrigeration is still required. If sample is acid preserved, results will be in the form of total nitrate and nitrite.

9.0 Quality Control

9.1 It is recommended that each laboratory that uses this method be required to operate a formal quality assurance program (16.1). The minimum requirements of this program consist of an initial demonstration of laboratory capability and ongoing analyses of laboratory prepared water standards as a test of continued performance to assess accuracy and precision. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1.1 The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 9.2. The laboratory shall, on an ongoing basis, demonstrate through analysis of the ongoing precision and recovery sample that the analysis system is in control.

9.1.2 Accompanying QC for the determination of nitrate is required per analytical batch. An analytical batch is a set of samples processed during a contiguous 8-hour period. Each analytical batch must be accompanied by an ongoing precision and recovery sample (OPR), matrix spike sample (MS), and matrix spike duplicate sample (MSD) resulting in a minimum of four analyses (1 OPR, 1 sample, MS, and MSD).

9.2 Initial demonstration of laboratory capability.

9.2.1 To establish the ability to detect nitrate the analyst shall determine the MDL and method limit (ML) per the procedure in 40 CFR 136, Appendix B (16.2) using the apparatus, reagents, and standards that will be used in the practice of this method. An achieved MDL and ML less than or equal to the MDL in Section 13.0 is recommended prior to the practice of this method.

9.2.2 Prepare and measure seven replicates of the MDL standard according to the procedure beginning in Section 7.4.1.

9.3 Initial precision and recovery (IPR) - To establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations:

9.3.1 Prepare and measure four samples of the IPR standard according to the procedure beginning in Section 7.5.

9.3.2 Using the results of the set of four analyses, compute the average percent recovery (x) and the standard deviation of the percent recovery (s) for nitrate. Use the following equation for calculation of the standard deviation of the percent recovery:

$$s = \sqrt{\frac{\sum x^2 - \frac{\left(\sum x\right)^2}{n}}{n-1}}$$

where:

n = Number of samples
x = % recovery in each sample

9.3.2.1 Compare s and x with the corresponding limits for initial precision and recovery in Table 1. If s and x meet the acceptance criteria, system performance is acceptable and analysis of samples may begin. If, however, s exceeds the precision limit or x falls outside the range for recovery, system performance is unacceptable. In this event correct the problem, and repeat the test.

9.4 Ongoing precision and recovery (OPR) - To demonstrate that the analysis system is in control, and acceptable precision and accuracy is being maintained with each analytical batch, the analyst shall perform the following operations:

9.4.1 Prepare a precision and recovery standard with each analytical batch.

9.4.1.1 At the end of each analytical batch of samples, analyze a precision and recovery standard. If the recovery is within the acceptable range, measurement process is in control and analysis of samples may proceed. If, however, the recovery is not in the acceptable range,

the analytical process is not in control. In this event, correct the problem, re-analyze analytical batch, repeating the ongoing precision and recovery test.

9.4.1.2 The laboratory should add results that pass to IPR and previous OPR data and update QC charts to form a graphic representation of continued laboratory performance. The laboratory should also develop a statement of laboratory data quality for each analyte by calculating the average percent recovery (R) and the standard deviation of the percent recovery (sr). Express the accuracy as a recovery interval from R - 2sr to R + 2sr. For example, if R = 95% and sr = 5%, the accuracy is 85% to 105%.

9.4.1.3 Depending upon specific program requirements, field replicate spikes may be required to assess the precision and accuracy of the sampling and sample transporting techniques.

10.0 Calibration and Standardization

10.1 The Hach DR series spectrophotometers have a built-in calibration that is automatically used when the TNTplus nitrate vial is inserted into the instrument. No further initial calibration is required. However, the instruments have the capability of developing a user-calibration. See manufacturer's manual for instructions.

10.2 Calibration Verification

10.2.1 To verify that the instrument is measuring nitrate properly, analyze a 0.3 mg/L and 10.0 mg/L nitrate nitrogen standard. Results should be within 15 percent of the actual value. Perform this calibration verification daily while instrument is in use.

11.0 Procedure

11.1 Instrument Setup – follow the instrument manufacturer's instructions for instrument setup.

11.2 Sample Preparation – Insure that the sample pH is between 3 and 10. If the sample pH is out of this range, adjust the sample pH with base or acid as needed.

11.2.1 For LR TNT 835: Pipet 1.0 mL of sample into the reagent vial.
For HR TNT836: Pipet 0.2 mL of sample into the reagent vial.

11.3 Reaction

11.3.1 For LR TNT835: Pipet 0.2 mL of Solution A into the vial.
For HR TNT836: Pipe 1.0 mL of Solution A into the vial.

11.3.2 Cap and invert the reaction tube 2-3 times until no more streaks can be seen in the reaction tube solution.

11.3.3 React for 15 minutes.

11.4 Analysis

11.4.1 Wipe the vial and insert the prepared vial into the spectrophotometer. The instrument reads the barcode, then selects and performs the correct test. No zero is required. Results are in mg/L NO_3^--N

12.0 Data Analysis and Calculations

12.1 Nitrate concentration is calculated automatically against internal instrument calibration.

13.0 Method Performance

Performance of the method was demonstrated in multi-lab studies comparing the method against currently promulgated nitrate methods. The method was evaluated in low ionic strength (LIS) and high ionic strength (HIS) matrices as well as multiple geographically diverse finished drinking water samples obtained from both surface water and ground water sources.

Acceptance Criterion	Section	Limit
Method Detection Limit	9.2.1	0.05 mg/L NO_3^- N
Method Limit	9.2.1	0.20 mg/L NO_3^- N
Initial Recovery Range	9.3.1	95.4% - 102%
Initial Precision	9.3.1	1.3
Matrix Recovery Range	9.4.1	90.5 – 101%

14.0 Pollution Prevention

14.1 Follow guidelines in Section 15.

15.0 Waste Management

15.1 It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect air, water, and land by minimizing and control all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

15.2 For further information on waste management, consult "The Waste Management manual for Laboratory Personnel", and "Less is Better: Laboratory Chemical Management for Waste Reduction", both available from the American Society's Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington, D.C. 20036.

16.0 References

16.1 "Handbook of Analytical Quality Control in Water and Wastewater Laboratories," USEPA, EMSL-CI, Cincinnati, OH 45268, EPA-600-4-79-019, March 1979.

16.2 40 CFR 136, Appendix B.

16.3 "OSHA Safety and Health Standards, General Industry," (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976)

16.4 "Safety in Academic Chemistry Laboratories," American Chemical Society, Committee on Chemical Safety, 3rd Edition, 1979.

16.5 "Water Analysis Handbook," Hach Company, 5th Edition, 2008.

17.0 Tables

17.1 Acceptance Criteria for Performance tests – The QC performance criteria for this method was performed with a Hach Company DR2800 Spectrophotometer and TNTplus 835 Reagent.

Table 1. Initial Precision and Recovery Method Performance

IPR Concentration	Average Recovery (%)	Average Standard Deviation
5.0 mg/L NO_3^- N	98.6	1.3

Table 2. Minimum Method Limit Performance

MDL Test Concentration	MDL	Rounded ML
0.30 mg/L NO_3^- N	0.05	0.20

18.0 Glossary of Definitions and Purposes

The definitions and purposes are specified to this method but have been conformed to common usage as much as possible.

18.1 Units of weight and measure and their abbreviations

18.1.1 Symbols
°C: degrees Celsius

18.1.2 Alphabetical characters
mg/L: milligram per liter

18.2 Definitions, acronyms, and abbreviations

18.2.1 MDL: Method detection limit

18.2.2 ML: Method limit

18.2.3 IPR: Initial precision and recovery

18.2.4 OPR: On-going precision and recovery

18.2.5 MS: Matrix spike

18.2.6 MSD: Matrix spike duplicate

18.2.7 LIS: Low ionic strength

18.2.8 HIS: High ionic strength

Nitrate

DOC316.53.01070

Dimethylphenol Method

Method 10206

0.23 to 13.50 mg/L NO_3^-–N or 1.00 to 60.00 mg/L NO_3^- (LR)

TNTplus™ 835

Scope and application: For wastewater, drinking water, surface water and process water.

Test preparation

Instrument-specific information

Table 1 shows all of the instruments that have the program for this test. The table also shows the adapter and light shield requirements for the applicable instruments that can use TNTplus vials.

To use the table, select an instrument, then read across to find the applicable information for this test.

Table 1 Instrument-specific information for TNTplus vials

Instrument	Adapters	Light shield
DR 6000, DR 5000	—	—
DR 3900	—	LZV849
DR 3800, DR 2800	—	LZV646
DR 1900	9609900 or 9609800 (A)	—

Before starting

DR 3900, DR 3800, DR 2800: Install the light shield in Cell Compartment #2 before this test is started.
Review the safety information and the expiration date on the package.
The recommended sample pH is 3–10.
The sample temperature must be 20–23 °C (68–73 °F) for accurate results.
The recommended temperature for reagent storage is 15–25 °C (59–77 °F).
DR 1900: Go to All Programs>LCK or TNTplus Methods>Options to select the TNTplus number for the test. Other instruments automatically select the method from the barcode on the vial.
Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.
Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

Items to collect

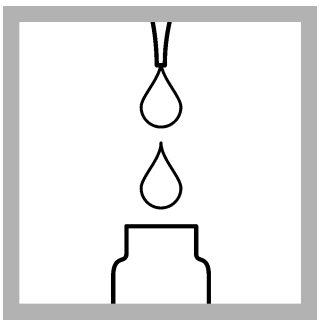
Description	Quantity
Nitrate LR TNTplus Reagent Set	1
Pipet, adjustable volume, 0.2–1.0 mL	1
Pipet tips, for 0.2–1.0 mL pipet	1

Refer to Consumables and replacement items on page 4 for order information.

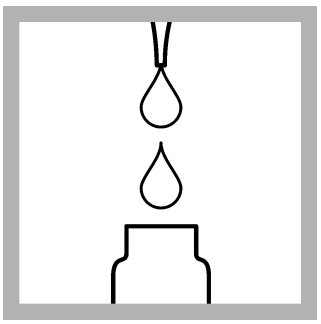
Sample collection and storage

- Collect samples in clean glass or plastic bottles.
- Analyze the samples as soon as possible for best results.
- If immediate analysis is not possible, immediately filter and keep the samples at or below 6 °C (43 °F) for a maximum of 48 hours.
- To preserve samples for a maximum of 14 days, adjust the sample pH to 2 or less with concentrated sulfuric acid (approximately 2 mL per liter) and keep at or below 6 °C (43 °F). The test results then include nitrate and nitrite.
- Let the sample temperature increase to room temperature before analysis.
- Before analysis, adjust the pH to 7 with 5 N sodium hydroxide solution.
- Correct the test result for the dilution caused by the volume additions.

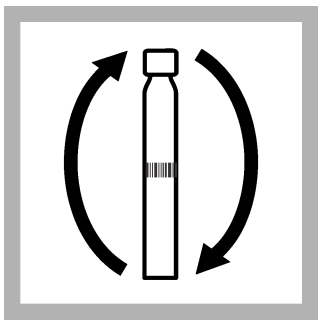
Test procedure



1. Use a pipet to add 1.0 mL of sample to the test vial.

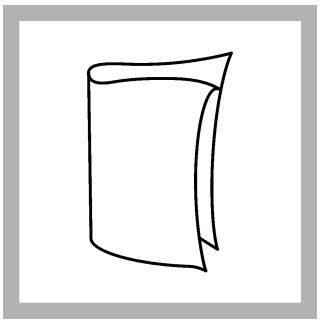


2. Use a pipet to add 0.2 mL of Solution A to the test vial.

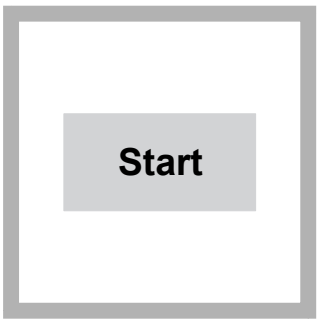


3. Tighten the cap on the vial and invert until completely mixed.

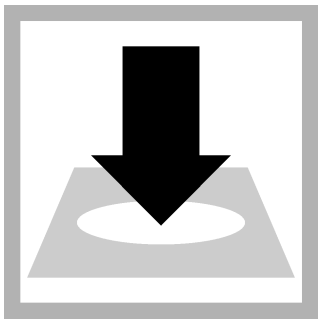
4. Start the reaction time of 15 minutes.



5. When the timer expires, clean the vial.



6. DR 1900 only: Select program 835. Refer to Before starting on page 1.



7. Insert the vial into the cell holder. DR 1900 only: Push **READ**.
Results show in mg/L NO_3^-–N.

Reagent blank correction

For the best results, measure the reagent blank value for each new lot of reagent. Replace the sample with deionized water in the test procedure to determine the reagent blank value. Subtract the reagent blank value from the sample results automatically with the reagent blank adjust option. Measure the reagent blank value when a new lot of reagent is used.

1. Use deionized water as the sample in the test procedure to measure the reagent blank value.
2. Set the reagent blank function to on. The measured reagent blank value is shown.

3. Accept the blank value. The reagent blank value is then subtracted from all results until the reagent blank function is set to off or a different method is selected.
Note: *As an alternative, record or enter the reagent blank value at a different time. Push the highlighted reagent blank box and use the keypad to enter the value.*

Sample blanks

If the sample has color or turbidity, measure a sample blank to correct the test result for the interference.
Items to collect:

- TNTplus™ 919 sample blank vial

1. Do the test procedure.
2. Put the sample in the sample blank vial. Fill to the neck of the sample blank vial.
3. Wipe the sample blank vial clean, then put it into the cell holder. If applicable, the instrument reads the barcode of the sample blank vial and subtracts the value from the initial test result.

Interferences

Table 2 shows that the ions were individually examined to the given concentrations and do not cause interference. No cumulative effects or influences of other ions were found.

The cumulative effects and influence of other ions have not been found. High loads of oxidizable organic substances (COD) cause the reagent to change color and to give high-bias results. The test can thus only be used for wastewater analyses if the COD is less than 500 mg/L. Verify measurement results with sample dilutions or standard additions.

Nitrite concentrations of more than 2.0 mg/L interfere (high-bias results). Add 50 mg of sulfamic acid (amidosulfonic acid) to 5.0 mL of sample, dissolve and wait for 10 minutes. Analyze the prepared sample as described in the procedure above.

Table 2 Interfering substances

Interfering substance	Interference level
Cl^-	500 mg/L
K^+	500 mg/L
Na^+	500 mg/L
Ca^{2+}	50 mg/L
Cd^{2+}	50 mg/L
Cu^{2+}	50 mg/L
Fe^{3+}	50 mg/L
Ni^{2+}	50 mg/L
Pb^{2+}	50 mg/L
Sn^{2+}	50 mg/L
Zn^{2+}	50 mg/L
Cr^{6+}	5 mg/L
NO_2^-	2 mg/L
Ag^+	100 mg/L
Co^{2+}	10 mg/L
Fe^{2+}	10 mg/L

Accuracy check

Standard solution method

Use the standard solution method to validate the test procedure, the reagents and the instrument.

Items to collect:

- Nitrate-Nitrogen Standard Solution, 10.0-mg/L NO_3^-–N or Wastewater Influent Standard Solution, Mixed Parameter

1. Use the test procedure to measure the concentration of the standard solution.
2. Compare the expected result to the actual result.

   Note: *The factory calibration can be adjusted slightly with the standard adjust option so that the instrument shows the expected value of the standard solution. The adjusted calibration is then used for all test results. This adjustment can increase the test accuracy when there are small variations in the reagents or instruments.*

Summary of Method

Nitrate ions in solutions that contain sulfuric and phosphoric acids react with 2,6-dimethylphenol to form 4-nitro-2,6-dimethylphenol. The measurement wavelength is 345 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
Nitrate LR TNTplus Reagent Set	1	25/pkg	TNT835

Required apparatus

Description	Quantity/test	Unit	Item no.
Pipet, adjustable volume, 0.2–1.0 mL	1	each	BBP078
Pipet tips, for 0.2–1.0 mL pipet	2	100/pkg	BBP079
Light shield, DR 3800, DR 2800, DR 2700	1	each	LZV646
Light shield, DR 3900	1	each	LZV849

Recommended standards

Description	Unit	Item no.
Nitrate Nitrogen Standard Solution, 10.0-mg/L NO_3–N	500 mL	30749
Nitrate Nitrogen Standard Solution 1000-mg/L NO_3–N	500 mL	1279249
Wastewater Influent Standard Solution, Mixed Parameter, for NH_3-N, NO_3-N, PO_4, COD, SO_4, TOC	500 mL	2833149

Optional reagents and apparatus

Description	Unit	Item no.
Filter membrane, 0.45-micron, 25-mm	100/pkg	2514101
Sampling bottle with cap, low density polyethylene, 500-mL	12/pkg	2087079
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	245032
Sulfamic Acid, 454 g	each	234401
Sulfuric Acid, concentrated, ACS	500 mL	97949

Optional reagents and apparatus (continued)

Description	Unit	Item no.
Test tube rack, polyethylene, for 13-mm OD vials, 90 holes	each	2497900
Water, deionized	4 L	27256

FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:
In the U.S.A. – **Call toll-free 800-227-4224**
Outside the U.S.A. – **Contact the HACH office or distributor serving you.**
On the Worldwide Web – **www.hach.com**; E-mail – **techhelp@hach.com**

HACH COMPANY
WORLD HEADQUARTERS
Telephone: (970) 669-3050
FAX: (970) 669-2932

09/2015, Edition 9

7. TURBIDITY

a. Turbidity Testing Walkthrough

Testing Methods – Turbidity

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Testing Procedures

Turbidity Testing

- Lamotte 2020 Turbidimeter with Turbidity tubes

Testing Procedures

First calibrate the Turbidity meter with a 10 NTU solution

Testing Procedures

After the Turbidimeter has been calibrated invert and mix the sample to be tested.

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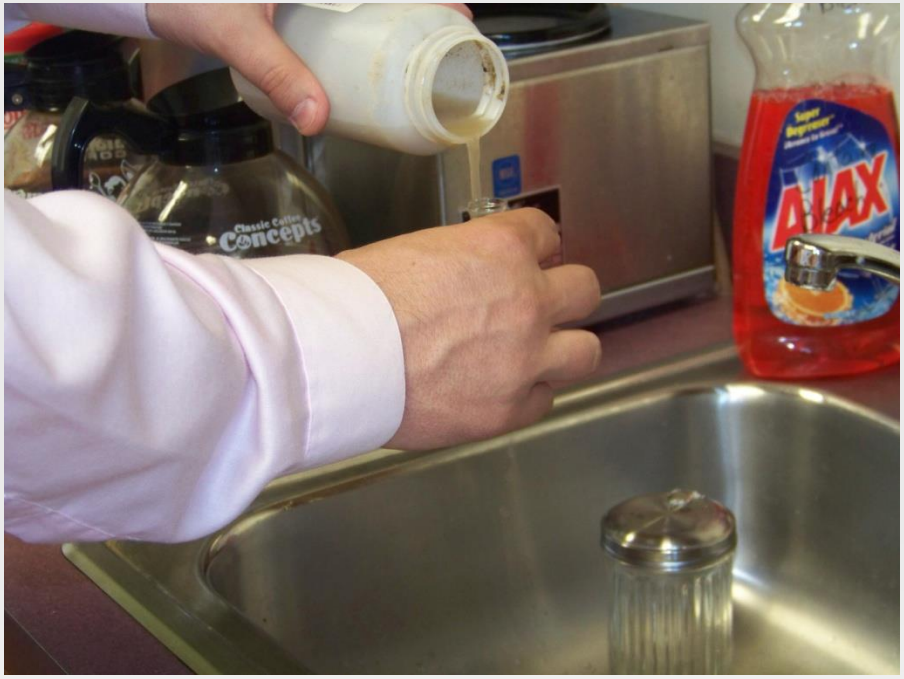
Testing Procedures



Pour sample into Turbidity tube.
Once sample is poured wipe down tube with a delicate task wiper.

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Testing Procedures

Insert Turbidity tube into meter with proper orientation and close lid

Testing Procedures

Once the lid is closed press the read button and wait for a reading to display.

After a reading, it may be a good idea to take the sample tube out of the Turbidimeter. Then invert the tube and re-insert in the Turbidimeter for another reading.

These readings can then be averaged and recorded.

b. EPA Method 180.1

United States Environmental Protection Agency

www.epa.gov August 1993

Method 180.1: Determination of Turbidity by Nephelometry

METHOD 180.1

DETERMINATION OF TURBIDITY BY NEPHELOMETRY

Edited by James W. O'Dell
Inorganic Chemistry Branch
Chemistry Research Division

Revision 2.0
August 1993

ENVIRONMENTAL MONITORING SYSTEMS LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268

METHOD 180.1

DETERMINATION OF TURBIDITY BY NEPHELOMETRY

1.0 SCOPE AND APPLICATION

1.1 This method covers the determination of turbidity in drinking, ground, surface, and saline waters, domestic and industrial wastes.

1.2 The applicable range is 0-40 nephelometric turbidity units (NTU). Higher values may be obtained with dilution of the sample.

2.0 SUMMARY OF METHOD

2.1 The method is based upon a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension. The higher the intensity of scattered light, the higher the turbidity. Readings, in NTU's, are made in a nephelometer designed according to specifications given in Sections 6.1 and 6.2. A primary standard suspension is used to calibrate the instrument. A secondary standard suspension is used as a daily calibration check and is monitored periodically for deterioration using one of the primary standards.

2.1.1 Formazin polymer is used as a primary turbidity suspension for water because it is more reproducible than other types of standards previously used for turbidity analysis.

2.1.2 A commercially available polymer primary standard is also approved for use for the National Interim Primary Drinking Water Regulations. This standard is identified as AMCO-AEPA-1, available from Advanced Polymer Systems.

3.0 DEFINITIONS

3.1 **Calibration Blank (CB)** -- A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes, internal standards, or surrogates analytes.

3.2 **Instrument Performance Check Solution (IPC)** -- A solution of one or more method analytes, surrogates, internal standards, or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.

3.3 **Laboratory Reagent Blank (LRB)** -- An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method

analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

3.4 **Linear Calibration Range (LCR)** -- The concentration range over which the instrument response is linear.

3.5 **Material Safety Data Sheet (MSDS)** -- Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

3.6 **Primary Calibration Standard (PCAL)** -- A suspension prepared from the primary dilution stock standard suspension. The PCAL suspensions are used to calibrate the instrument response with respect to analyte concentration.

3.7 **Quality Control Sample (QCS)** -- A solution of the method analyte of known concentrations that is used to fortify an aliquot of LRB matrix. The QCS is obtained from a source external to the laboratory, and is used to check laboratory performance.

3.8 **Secondary Calibration Standards (SCAL)** -- Commercially prepared, stabilized sealed liquid or gel turbidity standards calibrated against properly prepared and diluted formazin or styrene divinylbenzene polymers.

3.9 **Stock Standard Suspension (SSS)** -- A concentrated suspension containing the analyte prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source. Stock standard suspension is used to prepare calibration suspensions and other needed suspensions.

4.0 INTERFERENCES

4.1 The presence of floating debris and coarse sediments which settle out rapidly will give low readings. Finely divided air bubbles can cause high readings.

4.2 The presence of true color, that is the color of water which is due to dissolved substances that absorb light, will cause turbidities to be low, although this effect is generally not significant with drinking waters.

4.3 Light absorbing materials such as activated carbon in significant concentrations can cause low readings.

5.0 SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable.

5.2 Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in

this method. A reference file of Material Safety Data Sheets (MSDS) should be made available to all personnel involved in the chemical analysis. The preparation of a formal safety plan is also advisable.

5.3 Hydrazine Sulfate (Section 7.2.1) is a carcinogen. It is highly toxic and may be fatal if inhaled, swallowed, or absorbed through the skin. Formazin can contain residual hydrazine sulfate. Proper protection should be employed.

6.0 EQUIPMENT AND SUPPLIES

6.1 The turbidimeter shall consist of a nephelometer, with light source for illuminating the sample, and one or more photo-electric detectors with a readout device to indicate the intensity of light scattered at right angles to the path of the incident light. The turbidimeter should be designed so that little stray light reaches the detector in the absence of turbidity and should be free from significant drift after a short warm-up period.

6.2 Differences in physical design of turbidimeters will cause differences in measured values for turbidity, even though the same suspension is used for calibration. To minimize such differences, the following design criteria should be observed:

6.2.1 Light source: Tungsten lamp operated at a color temperature between 2200-3000°K.

6.2.2 Distance traversed by incident light and scattered light within the sample tube: Total not to exceed 10 cm.

6.2.3 Detector: Centered at 90° to the incident light path and not to exceed ±30° from 90°. The detector, and filter system if used, shall have a spectral peak response between 400 nm and 600 nm.

6.3 The sensitivity of the instrument should permit detection of a turbidity difference of 0.02 NTU or less in waters having turbidities less than 1 unit. The instrument should measure from 0-40 units turbidity. Several ranges may be necessary to obtain both adequate coverage and sufficient sensitivity for low turbidities.

6.4 The sample tubes to be used with the available instrument must be of clear, colorless glass or plastic. They should be kept scrupulously clean, both inside and out, and discarded when they become scratched or etched. A light coating of silicon oil may be used to mask minor imperfections in glass tubes. They must not be handled at all where the light strikes them, but should be provided with sufficient extra length, or with a protective case, so that they may be handled. Tubes should be checked, indexed and read at the orientation that produces the lowest background blank value.

6.5 Balance -- Analytical, capable of accurately weighing to the nearest 0.0001 g.

6.6 Glassware -- Class A volumetric flasks and pipets as required.

7.0 REAGENTS AND STANDARDS

7.1 Reagent water, turbidity-free: Pass deionized distilled water through a 0.45µ pore size membrane filter, if such filtered water shows a lower turbidity than unfiltered distilled water.

7.2 Stock standard suspension (Formazin):

7.2.1 Dissolve 1.00 g hydrazine sulfate, $(NH_2)_2.H_2SO_4$ (CASRN 10034-93-2) in reagent water and dilute to 100 mL in a volumetric flask. **CAUTION**-- carcinogen.

7.2.2 Dissolve 10.00 g hexamethylenetetramine (CASRN 100-97-0) in reagent water and dilute to 100 mL in a volumetric flask. In a 100 mL volumetric flask, mix 5.0 mL of each solution (Sections 7.2.1 and 7.2.2). Allow to stand 24 hours at 25 ±3°C, then dilute to the mark with reagent water.

7.3 Primary calibration standards: Mix and dilute 10.00 mL of stock standard suspension (Section 7.2) to 100 mL with reagent water. The turbidity of this suspension is defined as 40 NTU. For other values, mix and dilute portions of this suspension as required.

7.3.1 A new stock standard suspension (Section 7.2) should be prepared each month. Primary calibration standards (Section 7.3) should be prepared daily by dilution of the stock standard suspension.

7.4 Formazin in commercially prepared primary concentrated stock standard suspension (SSS) may be diluted and used as required. Dilute turbidity standards should be prepared daily.

7.5 AMCO-AEPA-1 Styrene Divinylbenzene polymer primary standards are available for specific instruments and require no preparation or dilution prior to use.

7.6 Secondary standards may be acceptable as a daily calibration check, but must be monitored on a routine basis for deterioration and replaced as required.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with turbidity free water. Volume collected should be sufficient to insure a representative sample, allow for replicate analysis (if required), and minimize waste disposal.

8.2 No chemical preservation is required. Cool sample to 4°C.

8.3 Samples should be analyzed as soon as possible after collection. If storage is required, samples maintained at 4°C may be held for up to 48 hours.

9.0 QUALITY CONTROL

9.1 Each laboratory using this method is required to operate a formal quality control (QC) program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and analysis of laboratory reagent blanks and other solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of data generated.

9.2 INITIAL DEMONSTRATION OF PERFORMANCE.

9.2.1 The initial demonstration of performance is used to characterize instrument performance (determination of LCRs and analysis of QCS).

9.2.2 Linear Calibration Range (LCR) -- The LCR must be determined initially and verified every six months or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to insure that the resulting curve is linear. The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by ±10%, linearity must be reestablished. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

9.2.3 Quality Control Sample (QCS) -- When beginning the use of this method, on a quarterly basis or as required to meet data-quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analysis of a QCS. If the determined concentrations are not within ±10% of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before continuing with on-going analyses.

9.3 ASSESSING LABORATORY PERFORMANCE

9.3.1 Laboratory Reagent Blank (LRB) -- The laboratory must analyze at least one LRB with each batch of samples. Data produced are used to assess contamination from the laboratory environment.

9.3.2 Instrument Performance Check Solution (IPC) -- For all determinations, the laboratory must analyze the IPC (a mid-range check standard) and a calibration blank immediately following daily calibration, after every tenth sample (or more frequently, if required) and at the end of the sample run. Analysis of the IPC solution and calibration blank immediately following calibration must verify that the instrument is

within ±10% of calibration. Subsequent analyses of the IPC solution must verify the calibration is still within ±10%. If the calibration cannot be verified within the specified limits, reanalyze the IPC solution. If the second analysis of the IPC solution confirms calibration to be outside the limits, sample analysis must be discontinued, the cause determined and/or in the case of drift the instrument recalibrated. All samples following the last acceptable IPC solution must be reanalyzed. The analysis data of the calibration blank and IPC solution must be kept on file with the sample analyses data. NOTE: Secondary calibration standards (SS) may also be used as the IPC.

9.3.3 Where additional reference materials such as Performance Evaluation samples are available, they should be analyzed to provide additional performance data. The analysis of reference samples is a valuable tool for demonstrating the ability to perform the method acceptably.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Turbidimeter calibration: The manufacturer's operating instructions should be followed. Measure standards on the turbidimeter covering the range of interest. If the instrument is already calibrated in standard turbidity units, this procedure will check the accuracy of the calibration scales. At least one standard should be run in each instrument range to be used. Some instruments permit adjustments of sensitivity so that scale values will correspond to turbidities. Solid standards, such as those made of lucite blocks, should never be used due to potential calibration changes caused by surface scratches. If a pre-calibrated scale is not supplied, calibration curves should be prepared for each range of the instrument.

11.0 PROCEDURE

11.1 Turbidities less than 40 units: If possible, allow samples to come to room temperature before analysis. Mix the sample to thoroughly disperse the solids. Wait until air bubbles disappear then pour the sample into the turbidimeter tube. Read the turbidity directly from the instrument scale or from the appropriate calibration curve.

11.2 Turbidities exceeding 40 units: Dilute the sample with one or more volumes of turbidity-free water until the turbidity falls below 40 units. The turbidity of the original sample is then computed from the turbidity of the diluted sample and the dilution factor. For example, if 5 volumes of turbidity-free water were added to 1 volume of sample, and the diluted sample showed a turbidity of 30 units, then the turbidity of the original sample was 180 units.

11.2.1 Some turbidimeters are equipped with several separate scales. The higher scales are to be used only as indicators of required dilution volumes to reduce readings to less than 40 NTU.

Note: Comparative work performed in the Environmental Monitoring Systems Laboratory - Cincinnati (EMSL-Cincinnati) indicates a progressive error on sample turbidities in excess of 40 units.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Multiply sample readings by appropriate dilution to obtain final reading.

12.2 Report results as follows:

NTU	Record to Nearest:
0.0 - 1.0	0.05
1 - 10	0.1
10 - 40	1
40 - 100	5
100 - 400	10
400 - 1000	50
>1000	100

13.0 METHOD PERFORMANCE

13.1 In a single laboratory (EMSL-Cincinnati), using surface water samples at levels of 26, 41, 75, and 180 NTU, the standard deviations were ±0.60, ±0.94, ±1.2, and ±4.7 units, respectively.

13.2 The interlaboratory precision and accuracy data in Table 1 were developed using a reagent water matrix. Values are in NTU.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 The quantity of chemicals purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

14.3 For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction," available from the American

Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington D.C. 20036, (202)872-4477.

15.0 WASTE MANAGEMENT

15.1 The U.S. Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water and land by minimizing and controlling all releases from hoods, and bench operations, complying with the letter and spirit of any waste discharge permit and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel," available from the American Chemical Society at the address listed in Section 14.3.

16.0 REFERENCES

1. Annual Book of ASTM Standards, Volume 11.01 Water (1), Standard D1889-88A, p. 359, (1993).

2. Standard Methods for the Examination of Water and Wastewater, 18th Edition, pp. 2-9, Method 2130B, (1992).

17.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

TABLE 1. INTERLABORATORY PRECISION AND ACCURACY DATA

Number of Values Reported	True Value (T)	Mean (X)	Residual for X	Standard Deviation (S)	Residual for S
373	0.450	0.4864	0.0027	0.1071	-0.0078
374	0.600	0.6026	-0.0244	0.1048	-0.0211
289	0.65	0.6931	0.0183	0.1301	0.0005
482	0.910	0.9244	0.0013	0.2512	0.1024
484	0.910	0.9919	0.0688	0.1486	-0.0002
489	1.00	0.9405	-0.0686	0.1318	-0.0236
640	1.36	1.3456	-0.0074	0.1894	0.0075
487	3.40	3.2616	-0.0401	0.3219	-0.0103
288	4.8	4.5684	-0.0706	0.3776	-0.0577
714	5.60	5.6984	0.2952	0.4411	-0.0531
641	5.95	5.6026	-0.1350	0.4122	-0.1078

REGRESSIONS: $X = 0.955T + 0.54$, $S = 0.074T + 0.082$

3. INFILTRATION TESTING

A. Infiltrometer Operating Instruction

Infiltrometer Operating Instructions

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Infiltrometer Operating Instructions

Setting the timer: Press the stop/reset button once to reset the timer to read “00 00”. Set the timer for 15 minutes.

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Infiltrometer Operating Instructions

Place double ring cutting blades on the area to be tested.

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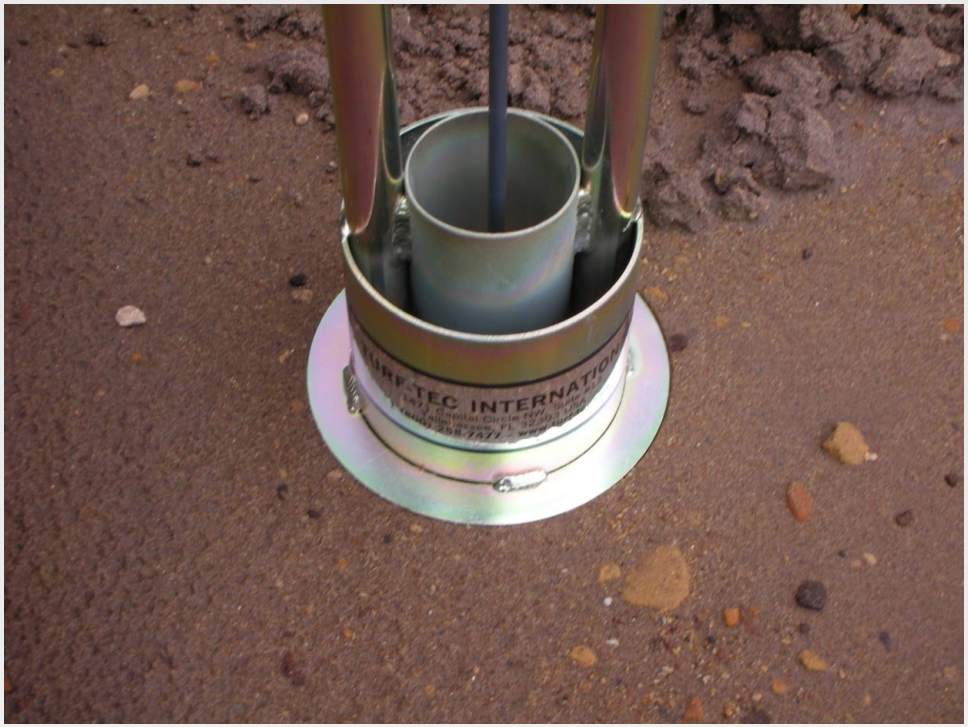
Infiltrometer Operating Instructions



Push down on the handle grips while slightly turning instrument back and forth until the saturation ring is against the soil surface.

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Infiltrometer Operating Instructions

Gently pour water into the inner ring and allow it to overflow to fill the outer ring. Once both rings are filled now measurements can be taken.

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Infiltrometer Operating Instructions

When the pointer reaches the beginning of the inch scale after filling both rings, start the timer immediately by pressing the start button.

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Infiltrometer Operating Instructions

As the water infiltrates into the soil, the plastic ball attached to the tube will measure the water in inches and register it on the scale with the pointer.

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Infiltrometer Operating Instructions

After 15 minutes the timer will beep. Record the number of inches according the scale at the 15 minute mark.

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Infiltrometer Operating Instructions

The first measurement observed is the **initial Infiltration rate**. Now repeat the previous steps filling the rings 2-3 times allowing the water to infiltrate into the soil between fillings. Once the soil is saturated, you can perform the tests. Continue to record measurements until the drop in water level is the same for two consecutive measurements. This will be the **basic infiltration rate**.

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Infiltrometer Operating Instructions

If the infiltration rate is slow, then a thirty minute or hour long test may be needed. If the infiltration rate is fast then a 2 or 5 minute test may be sufficient.

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Infiltrometer Operating Instructions

To remove the instrument from the soil, use the hand grips to rotate the cups in a twisting motion.

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Infiltrometer Operating Instructions

If any soil is removed use the plug pusher to remove the soil.

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Infiltrometer Operating Instructions

It may be best to take several readings on an area to get the average infiltration rate.

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