

# PROBE TECHNOLOGY AND LAB EQUIPMENT

OTCO October 13<sup>th</sup>, 2022

Ted Simmons – RSM – S. Ohio



# OVERVIEW OF HACH COMPANY

- **Began in 1947 in Ames, IA**
- **1999: Acquired by Danaher Corporation**
- **Manufacturer of industrial and municipal water analysis solutions**
  - process and laboratory instruments
  - chemistries
  - service and software
- **Innovation leader**
  - 527 patents, 130 patent families
  - strong investment in R&D and acquired technologies



- **OUR MISSION**

Ensure water quality for people around the world.

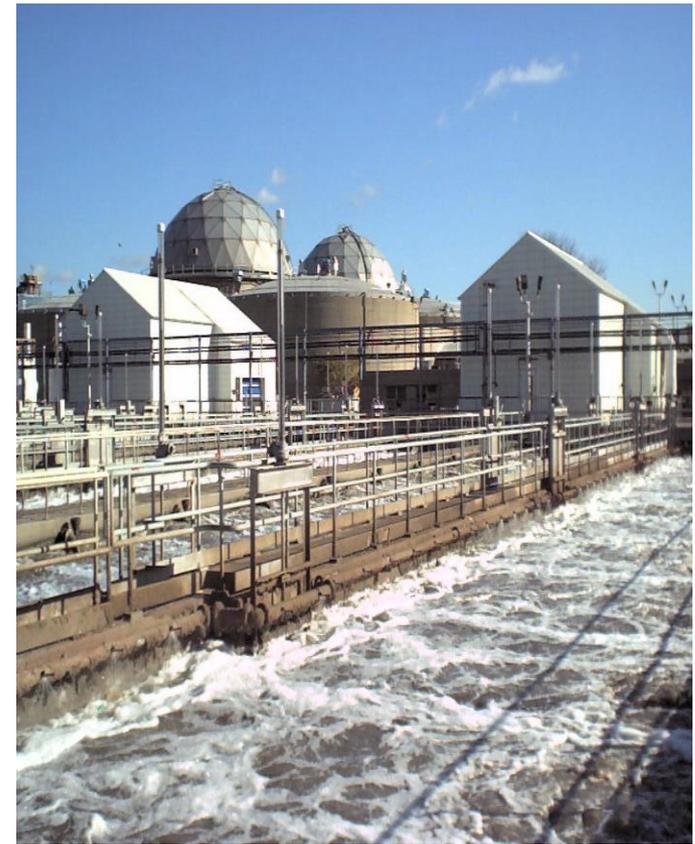
- **OUR VISION**

We make water analysis better—faster, simpler, greener and more informative—via unsurpassed customer partnerships, the most knowledgeable experts, and reliable, easy-to-use products.



# OVERVIEW OF HACH COMPANY

- **70+ RSM's**
  - Regional Sales Managers
- **25+ CAM - KAM – ADM – TSS - CSS**
  - CAM - Corporate Account Managers
  - KAM - Key Account Managers
  - ADM - Application Development Managers
  - TSS - Technical Sales Specialist
  - CSS - Complex Sales Specialist
- **100+ Field Service Technicians**
  - Field Service Partnerships
  - Startups, Commissioning and Training
- **41+ Technical Support Representatives**
  - Global Customer Support
- **12+ Bench Service Technicians**
  - Two service centers for repair and certification



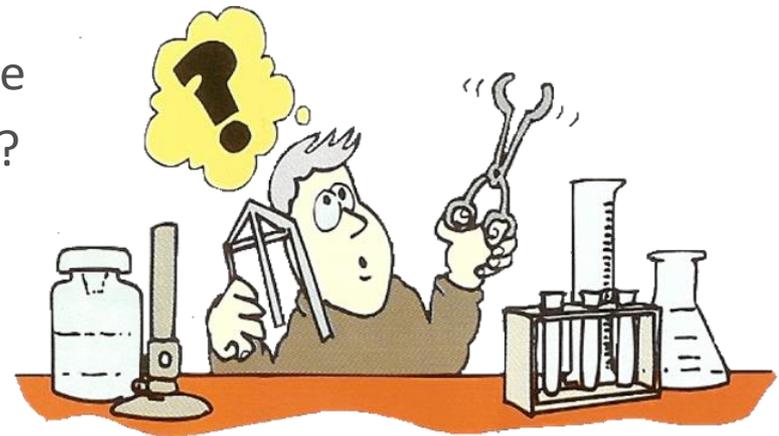
# ANALYTICAL PARAMETERS:

- pH
- Fluoride
- Conductivity
- Ammonia
- ORP
- Nitrate
- Dissolved Oxygen

# SELECTING THE RIGHT METHOD FOR YOUR LAB

Make sure that the procedure is correct for...

- Analyte
  - Nitrate vs. Nitrite
  - Total Phosphorus v. orthophosphate
  - Interferences particular to method?
- Concentration range
  - What is my expected result?
  - ppb vs. ppm
- Is this for compliance reporting?
  - Does it need regulatory approval?



# ON-SITE TESTING

- Field Testing
  - pH/Temp
  - Chlorine (Within 15 minutes)
  - **Dissolved Oxygen**
- **These parameters MUST be tested on site.**
  - You can't send them out!!!
- NO sample preservation is allowed.
- Properly log the data.

# FIELD OR LAB?



# PROBE STYLE LAB PARAMETERS

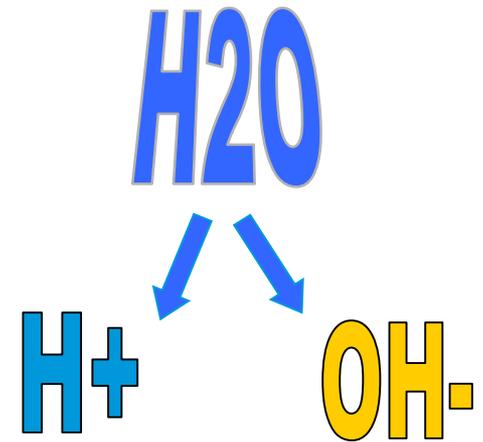
## *Parameter*

- Temperature
- pH
- Conductivity
- Dissolved Oxygen
- Luminescent (LDO)
- BOD Sensor (with LDO)
- ORP/Redox
- Ion Selective Electrodes
  - Ammonia
  - Fluoride
  - Nitrate



# PH THEORY

- pH is a measurement of the relative acidity of an aqueous solution
- pH is a measurement of hydrogen ion concentration
- Acid - increases the hydrogen ion ( $H^+$ ) concentration in a solution
- Base - increases the hydroxide ion ( $OH^-$ ) concentration in a solution



# PH THEORY

- pH is defined as the negative log of the molar hydrogen ion concentration in aqueous solution

$$\text{pH} = -\log [\text{H}^+]$$

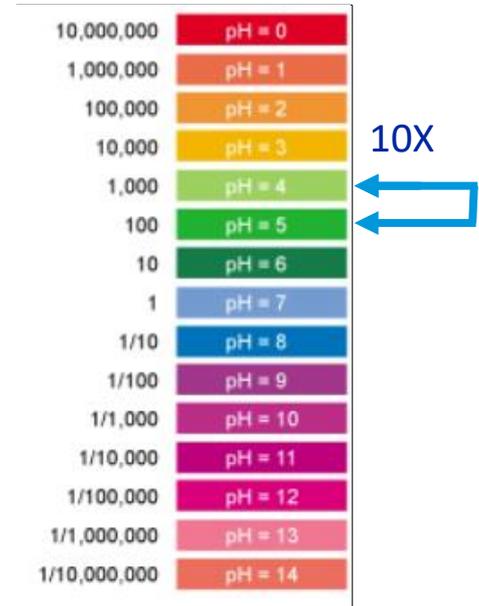
# PH SCALE

Concentration of hydrogen ions compared to distilled water		Examples of solutions at this pH
10,000,000	pH = 0	battery acid, strong hydrofluoric acid
1,000,000	pH = 1	hydrochloric acid secreted by stomach lining
100,000	pH = 2	lemon juice, gastric acid, vinegar
10,000	pH = 3	grapefruit, orange juice, soda
1,000	pH = 4	tomato juice, acid rain
100	pH = 5	soft drinking water, black coffee
10	pH = 6	urine, saliva
1	pH = 7	"pure" water
1/10	pH = 8	sea water
1/100	pH = 9	baking soda
1/1,000	pH = 10	Great Salt Lake, milk of magnesia
1/10,000	pH = 11	ammonia solution
1/100,000	pH = 12	soapy water
1/1,000,000	pH = 13	bleaches, oven cleaner
1/10,000,000	pH = 14	liquid drain cleaner

The scale is courtesy of The Pacific Institute for the Mathematical Sciences

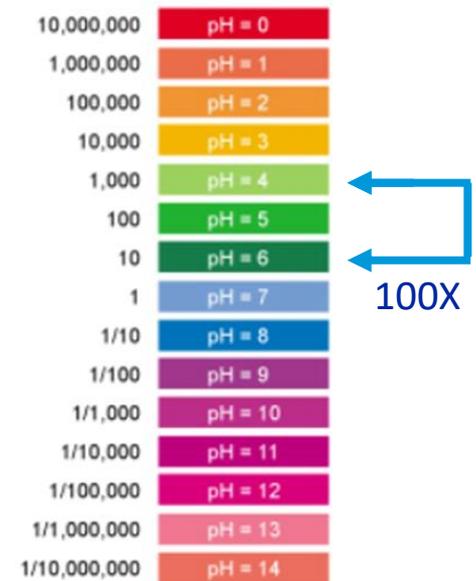
# PH SCALE

- pH is a negative logarithmic function
- Each decrease in pH unit = 10X increase in acidity
  - Solution at pH4 is 10X more acidic than solution at pH5



# PH SCALE

- pH is a negative logarithmic function
- Each decrease in pH unit = 10X increase in acidity
  - Solution at pH 4 is 100X more acidic than pH6 solution



# HOW DOES A PH PROBE WORK?

- Probe measures hydrogen ion concentration
  - Two electrodes in probe (combination electrodes)
    - sensing half-cell
    - reference half-cell
  - Third reference electrode (differential probes)

# HALF-CELLS

- Ion sensing pH half cell
  - Glass bulb that is sensitive to  $H^+$ .
- Reference half-cell
  - Glass tube filled with salt solution to complete circuit.

# METER

Ion Sensing Half-Cell

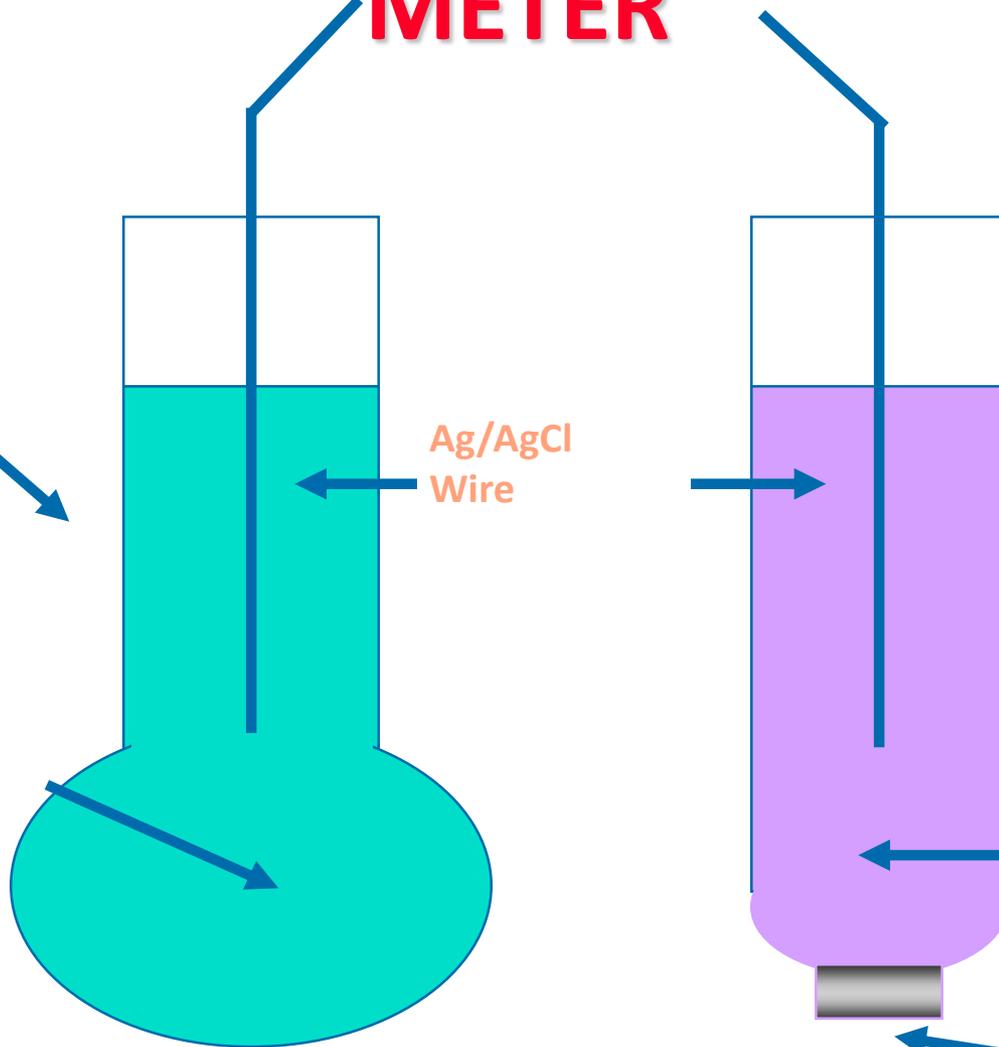
Reference Half-Cell

Ag/AgCl Wire

Internal Filling Solution

Reference Electrolyte

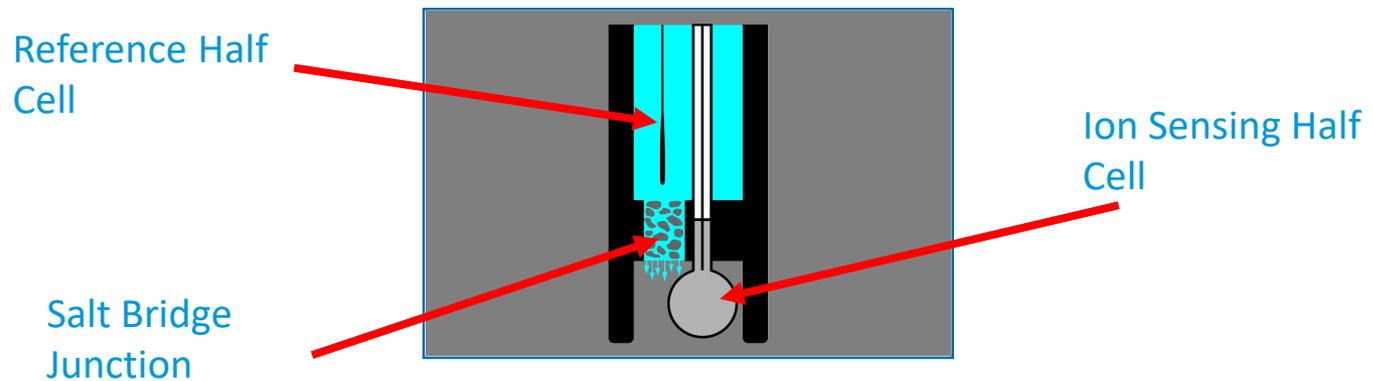
Salt Bridge Junction



Be Right™

# REFERENCE HALF-CELL

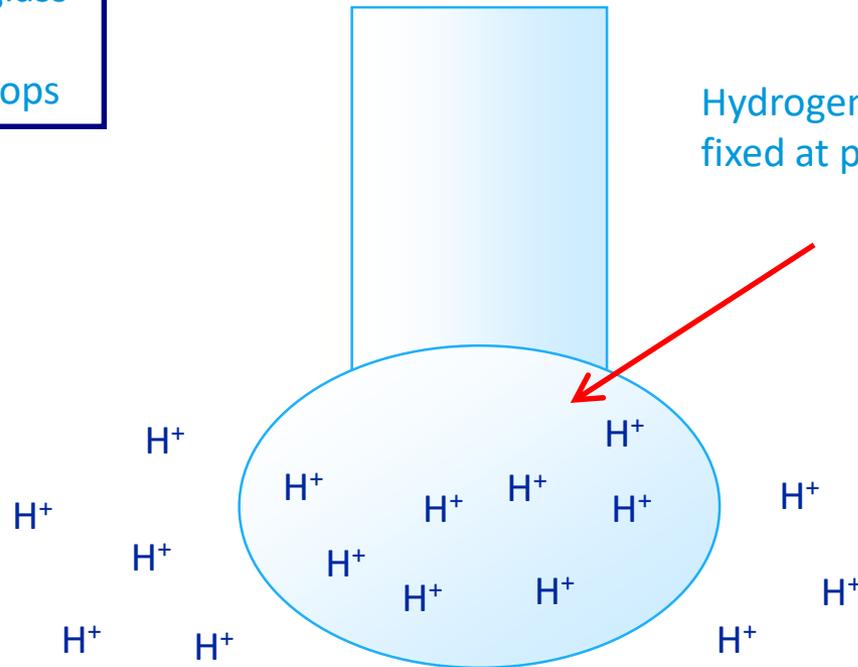
- Dispenses reference solution which completes circuit for meter



**pH 7 Solution**

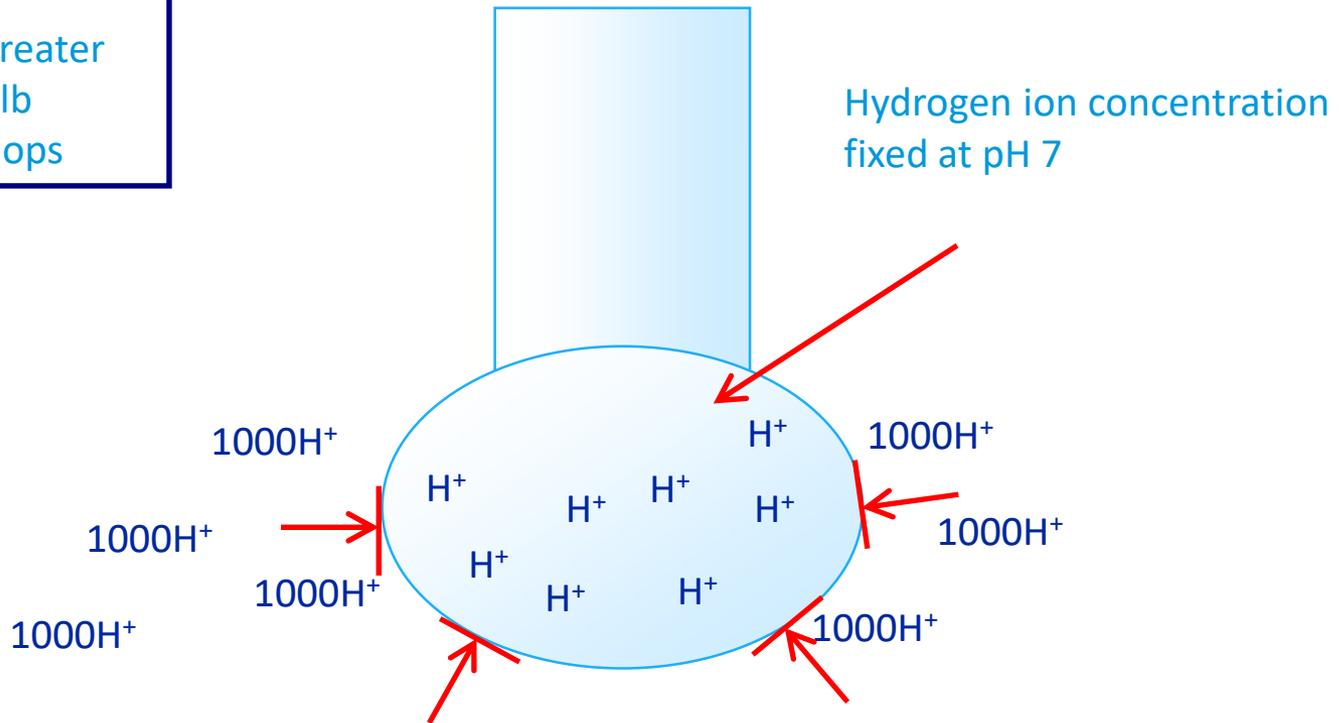
H<sup>+</sup> conc the same both  
inside and outside glass  
bulb

\*No potential develops



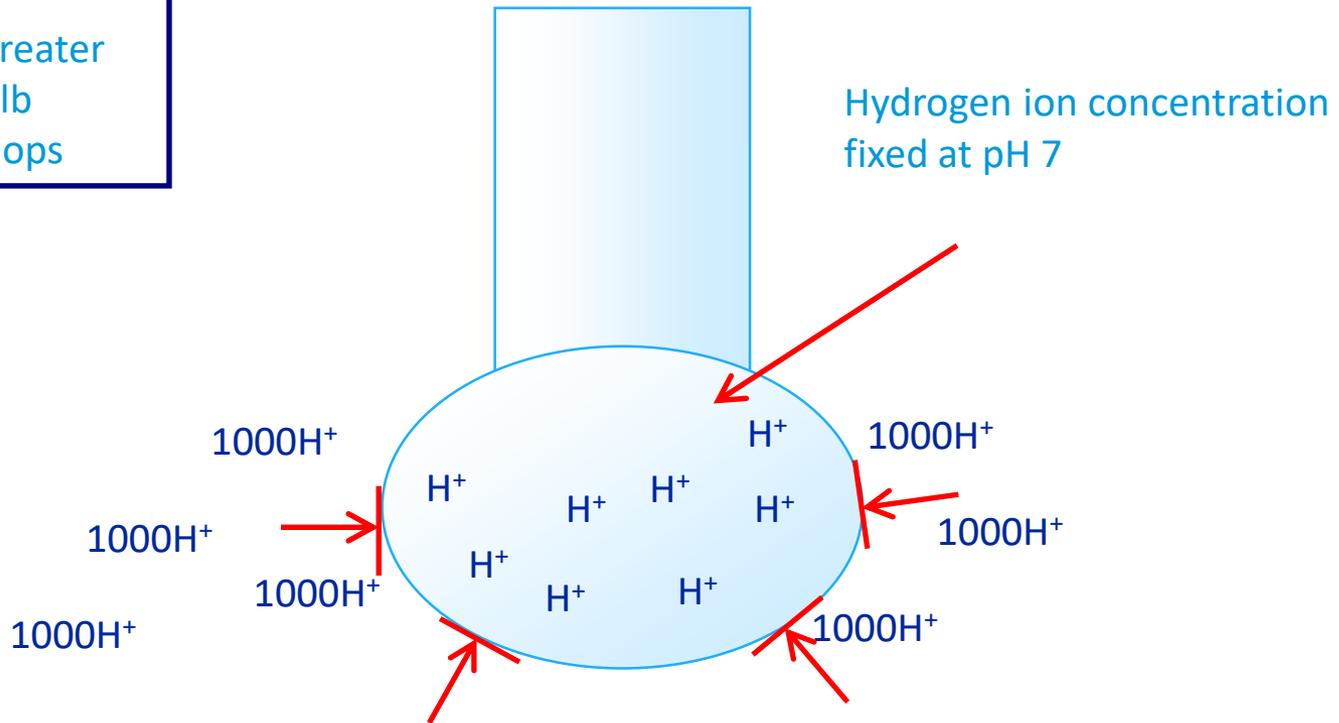
**pH 4 Solution**

H<sup>+</sup> conc 1000x greater  
outside glass bulb  
\*Potential develops



**pH 4 Solution**

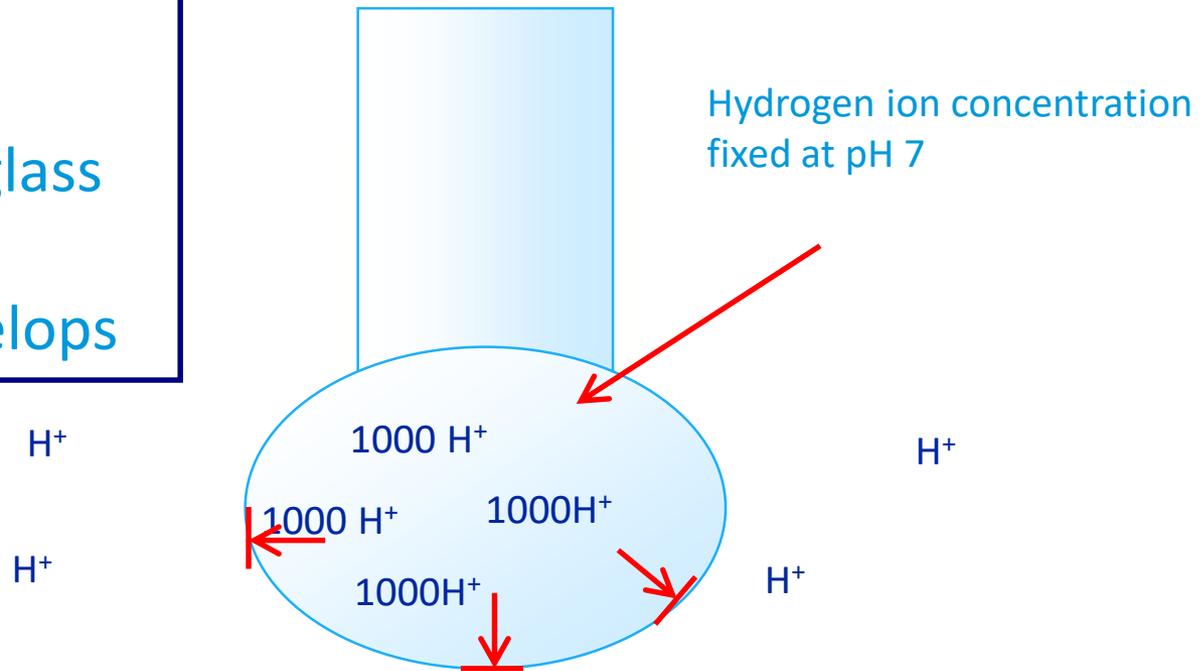
H<sup>+</sup> conc 1000x greater  
outside glass bulb  
\*Potential develops



## pH 10 Solution

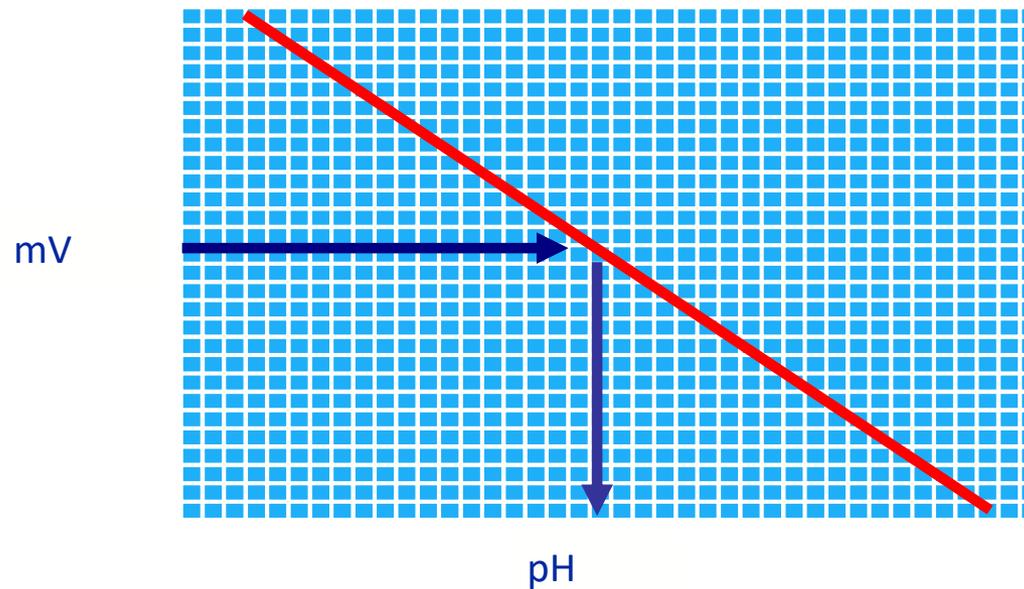
$H^+$  conc 1000x  
greater inside glass  
bulb

\*Potential develops

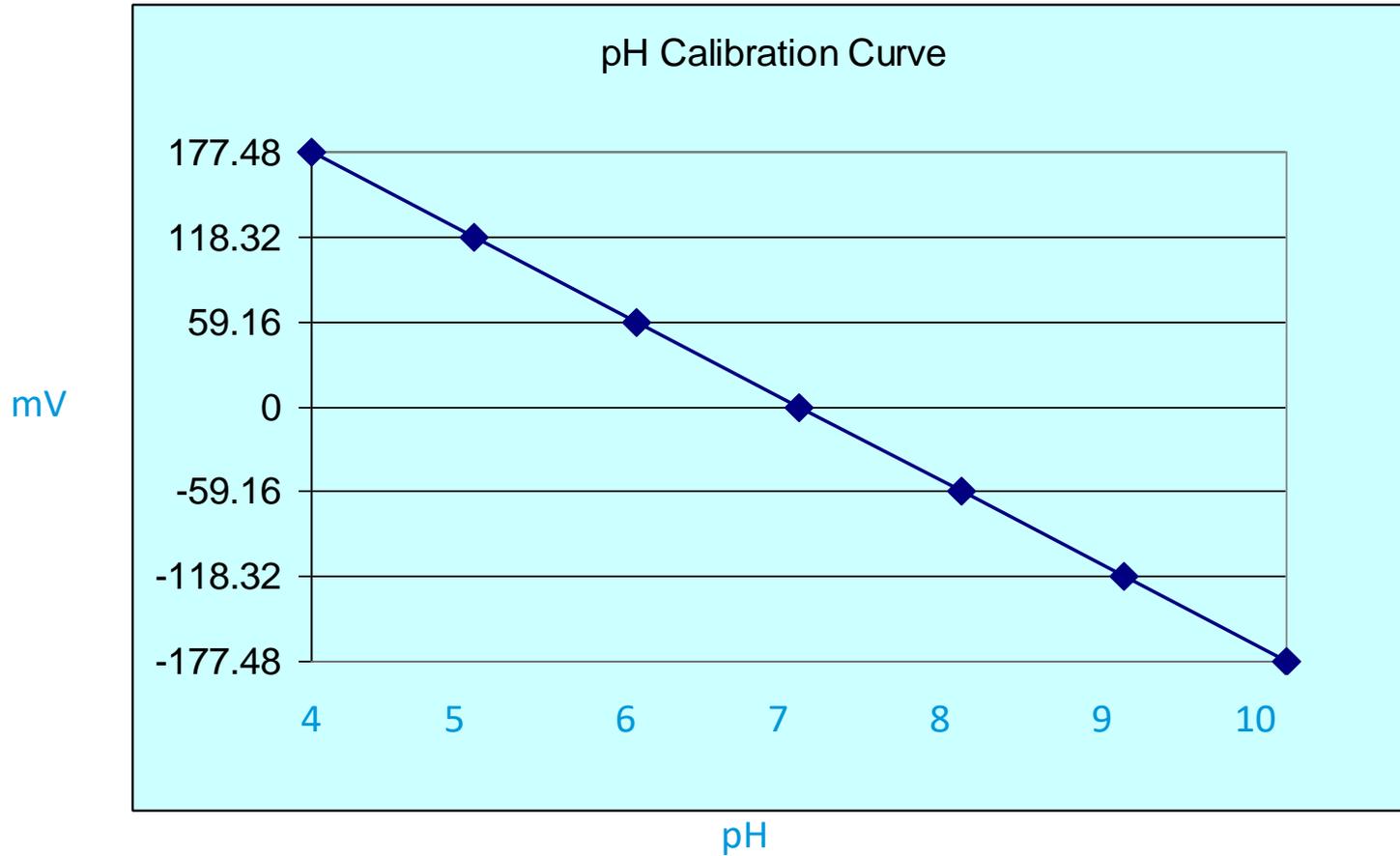


# CALIBRATION

- A calibration curve allows the meter to convert a measured millivolt potential into a pH reading.



# CALIBRATION



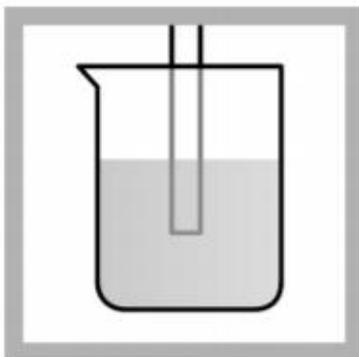
## Sample collection

- Analyze the samples immediately. The samples cannot be preserved for later analysis.
- Collect samples in clean glass or plastic bottles.

## Test procedure



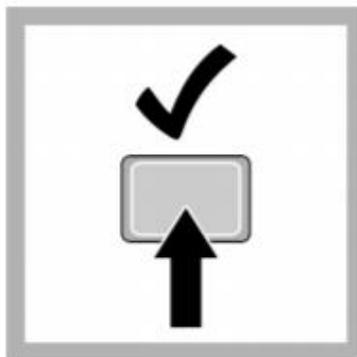
**1.** Rinse the probe with deionized water. Dry the probe with a lint-free cloth.



**2. Laboratory test:** Put the probe in a beaker that contains the solution. Do not let the probe touch the stir bar, bottom or sides of the container. Remove the air bubbles from under the probe tip. Stir the sample at a slow to moderate rate.

**Field test:** Put the probe in the sample. Move the probe up and down to remove bubbles from the electrode.

Make sure to put the temperature sensor fully in the sample.



**3.** Push **Read**. A progress bar is shown. When the measurement is stable, the lock icon is shown.



**4.** Rinse the probe with deionized water. Dry the probe with a lint-free cloth.

# STORAGE

- KCL probe storage solution
- pH4 buffer
- pH7 buffer
- NEVER use DI water for probe storage

# CLEANING

- Rinse with DI water
- Clean with mild detergent solution and soft cloth
- Alternately soak in 0.1 N hydrochloric acid and 0.1N sodium hydroxide solution for a few minutes and then soak in DI water for 15 minutes.
- NEVER use a stiff brush on a pH probe

# CALIBRATION FAILED, NOW WHAT?

- Repeat Calibration (Perfect Slope -59.18 mv/pH)
- Check Buffers (Date, Temperature) Did they sit in the truck all summer? Over the winter? For 2 years?
- Fresh buffers?
- Clean probe – Buffer 4, vinegar, Coca-Cola?
- Call the manufacturer

# HOW LONG WILL A PH PROBE LAST?

- Proper storage is everything
- Rinse with DI water before storage
- Store in pH Storage solution. (PN 2756549)
- Store in a vertical position



# KEEP IT CLEAN



Just because your pH reading never changes doesn't mean it's correct.



# ANALYTICAL PARAMETERS:

- pH
- **Fluoride**
- Conductivity
- Ammonia
- ORP
- Nitrate
- Dissolved Oxygen

# METHODS OF ANALYSIS

- **SPADNS Colorimetric**
- **Electrochemical ISE**

## Interferences in Fluoride Measurement\*

\*Standard Methods for the Examination of Water and Wastewater, 19th ed.

Substance	Electrode Method		SPADNS Method	
	Conc., mg/l	Error (+/-)	Conc., mg/l	Error (+/-)
Alkalinity as CaCO <sub>3</sub>	7,000	+	5,000	-
Aluminum (Al <sup>3+</sup> )	3	-	0.1	-
Chloride (Cl <sup>-</sup> )	20,000	*	7000	+
Chlorine	5000	*	5	Over 5 mg/l, add sodium arsenite
Color		*		Remove or compensate for
Turbidity		*		Remove or compensate for
Iron	200	-	10	-
Hexameta-phosphate ( [Na(PO <sub>3</sub> ) <sub>6</sub> ] )	50,000	*	1	+
Phosphate (PO <sub>4</sub> <sup>3-</sup> )	50,000	-	16	-
Sulfate (SO <sub>4</sub> <sup>2-</sup> )	50,000	-	200	-

\* No measureable error

- If an interference exceeds the limit shown, distillation is required for either method before measurement

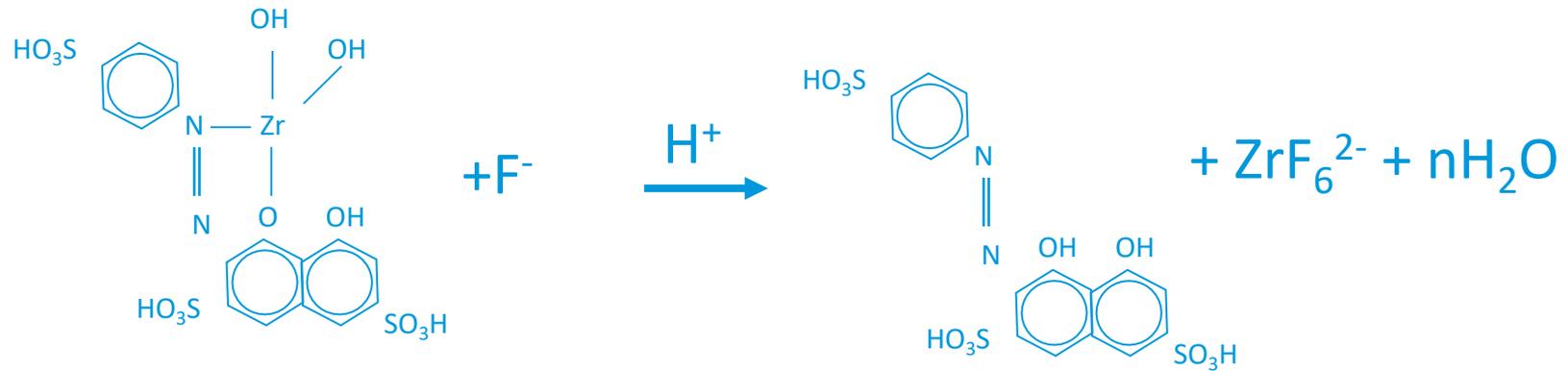
# ELECTROCHEMICAL METHOD

- Uses and Ion-Selective Electrode system
- Silver/silver chloride reference electrode
- Lanthanum fluoride (LaF)
- Utilizes TISAB
  - Total Ionic Strength Adjustment Buffer
  - Adjusts pH
  - Provides buffering
  - Complex interferences (Al, Fe)

# ADVANTAGES OF THE ISE METHOD

- Eliminates the need for most distillations
- Rapid
- New combination electrode
  - Eliminates tip replacement
  - Eliminates filling solution
  - No separate reference half-cell

# ARSENIC-FREE SPADNS 2 REAGENT



Zirconium-dye  
complex (Red)

Colorless

- Bleaching reaction, decrease in color intensity (absorbance) is measured at 580 nm
- Arsenic-free SPADNS 2 available in bottles or AccuVac Ampuls

# ANALYTICAL PARAMETERS:

- pH
- Fluoride
- **Conductivity**
- Ammonia
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- Nitrate
- Dissolved Oxygen

# CONDUCTIVITY



# WHAT IS CONDUCTIVITY?

A measure of the ability of a solution to conduct an electric current.

**\*Sometimes called “Specific Conductance”**

# WHAT IS RESISTIVITY?

A measure of the ability of a solution to resist an electric current.

# CONDUCTIVITY VS. RESISTIVITY

Conductivity (C) is the reciprocal of Resistivity (R)

$$C = 1/R$$

Pure water = 18.2 MΩ

$$C = 1/18.2 \text{ M}\Omega = 0.056\mu\text{S}$$

# WHY MEASURE CONDUCTIVITY ?

- As a measure of percent acid or caustic
- To measure contaminant rate
- To measure water purity
- To measure process efficiencies
- *Because you have to!*

# CONDUCTIVITY OF VARIOUS SOLUTIONS

<b>Theoretical pure water</b>	<b>0.056 microSiemens/cm</b>
<b>Demineralized water</b>	<b>0.056 to 1.0 microSiemens/cm</b>
<b>Reverse osmosis water</b>	<b>0.8 to 3.0 microSiemens/cm</b>
<b>Distilled water</b>	<b>1 to 10 microSiemens/cm</b>
<b>Tap water</b>	<b>100 to 2000 microSiemens/cm</b>
<b>Sea water</b>	<b>40,000 to 100,000 microSiemens/cm</b>
<b>5% Sodium chloride solution</b>	<b>70,000 microSiemens/cm</b>
<b>10% Sulfuric acid solution</b>	<b>140,000 microSiemens/cm</b>

# ANALYTICAL PARAMETERS:

- pH
- Fluoride
- Conductivity
- **Ammonia**
- ORP
- Nitrate
- Dissolved Oxygen

# MEASURING AMMONIA

## Ammonia probe - ISE



## TNT+ Reagents - Colorimetric

- Multiple Ranges



# ANALYTICAL PARAMETERS:

- pH
- Fluoride
- Conductivity
- Ammonia
- **ORP**
- Nitrate
- Dissolved Oxygen

# WHAT IS ORP?

- Oxidation Reduction Potential (Redox)
- Measure of the chemical equilibrium potential of a system
- Potential is generated by the relative concentration of chemical oxidants and reductants

# OXIDANTS AND REDUCTANTS

## Oxidant

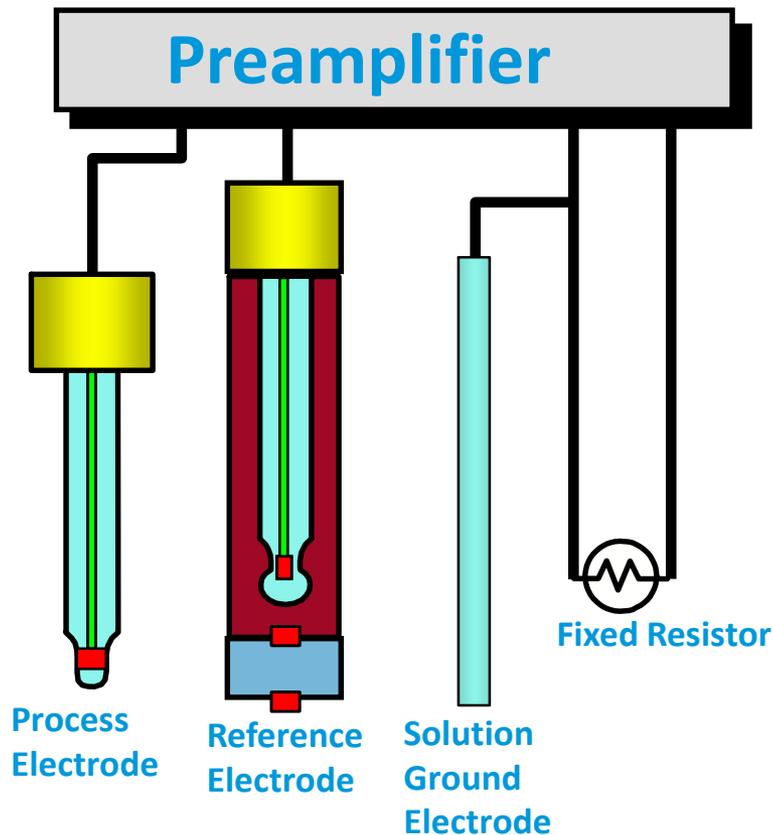
- Creates a more positive potential (electron acceptor)  
example: Chlorine, Bromine

## Reductant

- Creates a more negative potential (electron donor)  
example: Sodium bisulfate,  
Sodium hypochlorite

# ORP MEASUREMENT

## Differential Technique



$$\frac{E_1 - E_3}{-(E_2 - E_3)}$$

---

$$(E_1 - E_2)$$

# HACH COMBINATION ORP SENSORS



# ANALYTICAL PARAMETERS:

- pH
- Fluoride
- Conductivity
- Ammonia
- ORP
- **Nitrate**
- Dissolved Oxygen

# TESTING FOR NITRATES

## Nitrate Probe – ISE Style



## TNT+ Reagents - Colorimetric

- Multiple ranges



# ANALYTICAL PARAMETERS:

- pH
- Fluoride
- Conductivity
- Ammonia
- ORP
- Nitrate
- **Dissolved Oxygen**

# DISSOLVED OXYGEN TESTING

- Done with a probe on location!
- Cap should be replaced once a year
- Dry storage
- Verification can be performed
- **Do NOT use Accu-Vacs for Dissolved Oxygen – not approved for wastewater**



# FIELD TESTING DO

## Sample collection

The main consideration with sample collection is to prevent contamination of the sample with atmospheric oxygen.

- Analyze the samples immediately. The samples cannot be preserved for later analysis.
- Analyze the samples at the collection site if possible.
- Do not introduce air into the sample.

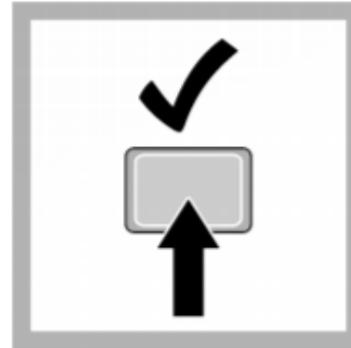
## Test procedure



**1.** Rinse the probe with deionized water. Dry the probe with a lint-free cloth.



**2. Laboratory test:** Put the probe in a beaker that contains the solution. Do not let the probe touch the stir bar, bottom or sides of the container. Remove the air bubbles from under the probe tip. Stir the sample at a slow to moderate rate.  
**Field test:** Put the probe in the sample. Move the probe up and down to remove bubbles from the probe tip.



**3. Push Read.** A progress bar is shown. When the measurement is stable, the lock icon is shown.



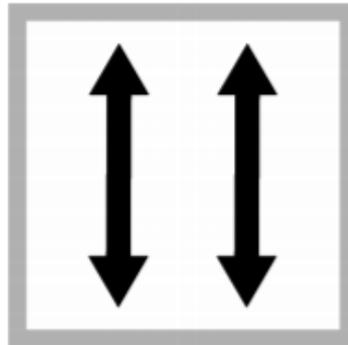
**4.** When the value is stable, store or record the mV value and the temperature value.

# CALIBRATION OF DO

## Calibration



1. Add a small amount of water (approximately 1 cm) to the bottom of narrow-neck bottle, such as a BOD bottle.



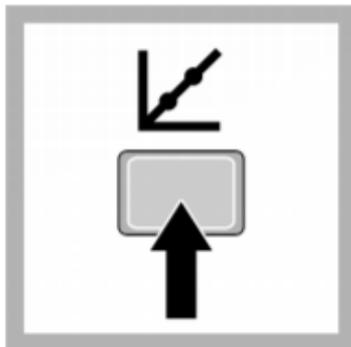
2. Insert a stopper and shake the bottle vigorously for several minutes.



3. If the probe cap is wet, carefully dry the probe cap with a soft cloth.



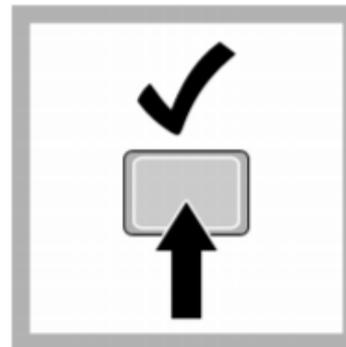
4. Remove the stopper. Put the probe in the bottle.



5. Push **Calibrate**.



6. Push **Read**. A progress bar is shown. When the measurement is stable, the lock icon is shown.



7. Push **Done**. The calibration summary shows. The slope value is the comparison between the latest calibration and the factory calibration shown as a percentage.



8. Push **Store** to accept the calibration.

# DISSOLVED OXYGEN TESTING

- Replace cap every couple years



# BENCHTOP SPECTROPHOTOMETER WITH RFID\* TECHNOLOGY

- VIS Spectrophotometer
- UV/VIS Spectrophotometer
  - Guided Procedures
  - Eliminate False Readings
  - Hands Free Updates
  - Flexible Connectivity
    - 1 Ethernet
    - 3 USB
  - Sample Tracking w/RFID



# WATER/ WASTEWATER LAB TESTING

- Can be done with these instruments
  - Pocket Colorimeter II, DR300 (One Parameter)
  - DR900 – Portable - Change the batteries once a year
  - DR3900 - Laboratory



# EPA COMPLIANT METHODS

EPA Methods

## Hach EPA Compliant Methods

### EPA-Approved Methods

The EPA has evaluated and approved new technological methods developed by Hach Company. All EPA-Approved methods are cited in the Federal Register and compiled in the Code of Federal Regulations at 40 CFR 136 and CFR 141.

### EPA-Accepted Methods

The EPA has reviewed Hach methods and accepted them for use in compliance monitoring. These methods are defined by EPA as Acceptable versions of previously approved methods. These methods are generally not published in the Federal Register or in the Code of Federal Regulations. A facsimile of the EPA- Acceptance letter is available upon request.

### EPA-Equivalent Methods

All EPA-Approved methods have specification criteria built into their procedural steps. When an approved or accepted EPA method has been packaged by Hach from the EPA reference method as a test method that meets or exceeds these specification criteria, these methods are deemed to be equivalent for use in EPA compliance monitoring (40 CFR 136.6). EPA does not normally issue equivalence letters of packaged reference methods. Hach maintains the formulation, procedure, and data demonstrating equivalency and is available upon request.

### Hach Approved Methods

These methods may be used for compliance monitoring. They have either obtained an EPA Approval or Acceptance letter, or the method is a packaged product that follows an EPA Reference method and is deemed Equivalent under 40 CFR 136.6. With any method used for compliance reporting, always consult with your local regulatory authority.



Analyte	Sample Matrix <sup>1</sup>	Hach Method	Approval Type	Reference Method	See Page
Acidity, as CaCO <sub>3</sub> , Phenolphthalein, Buret	WW	8010	Accepted	SM 2310 B	12
Acidity, Phenolphthalein, Digital Titration	WW	8202	Equivalent	SM 2310 B	12
Alkalinity, Digital Titration	WW	8203	Equivalent	SM 2320 B, EPA 310.2	14
Ammonia as Nitrogen	WW	TNT830	Equivalent	EPA 350.1, EPA 351.1, EPA 351.2	16
Ammonia as Nitrogen	WW	TNT831	Equivalent	EPA 350.1, EPA 351.1, EPA 351.2	16
Ammonia as Nitrogen	WW	TNT832	Equivalent	EPA 350.1, EPA 351.1, EPA 351.2	16
Ammonia Nitrogen, Electrode	WW	10001	Equivalent	SM 4500-NH <sub>3</sub> D, E, F, or G	108
Ammonia Nitrogen, Known Addition, Electrode	WW	10002	Equivalent	SM 4500-NH <sub>3</sub> D, E, F, or G	108
Ammonia, as Nitrogen, Nessler	WW	8038	Accepted	SM 4500-NH <sub>3</sub> C	16
Arsenic, Total	WW	8013	Accepted	SM 3500-As B or C	18
BOD	WW	8043	Accepted	SM 5210 B	20
Calcium, Total, Buret	DW	8222	Accepted	SM 3500-Ca B or D	22
Calcium, Total, Buret	WW	8222	Accepted	SM 3500-Ca B or D	22
Chemical Oxygen Demand	WW	8000	Approved	40 CFR 136.3	32
Chemical Oxygen Demand, TNTplus	WW	TNT821/ 8000	Approved	Hach Method 8000, 40 CFR 136.3	32
Chemical Oxygen Demand, TNTplus	WW	TNT822/ 8000	Approved	Hach Method 8000, 40 CFR 136.3	32
Chloride, Mercuric Nitrate, Digital Titration	WW	8206	Equivalent	SM 4500-Cl C	24
Chloride, Silver Nitrate, Buret	WW	8225	Accepted	SM 4500-Cl B	24
Chloride, Silver Nitrate, Digital Titration	WW	8207	Equivalent	SM 4500-Cl B	24
Chlorine, Free, DPD	DW	8021	Accepted	SM 4500-Cl G	25
Chlorine, Free Amperometric Titration	DW	8334	Equivalent	SM 4500-Cl D	26
Chlorine, Free Amperometric Titration	WW	8334	Equivalent	SM 4500-Cl D	26
Chlorine, Total, DPD ULR	DW	8370	Accepted	SM 4500-Cl G	25
Chlorine, Total Amperometric Forward Titration	DW	10026	Accepted	SM 4500-Cl D	26
Chlorine, Free, DPD MR	DW	10245	Accepted	SM 4500-Cl G	28
Chlorine, Free, DPD MR	WW	10245	Accepted	SM 4500-Cl G	28
Chlorine, Free, DPD HR	DW	10069	Accepted	SM 4500-Cl G	26

<sup>1</sup>DW = Drinking Water; SS = Sewage Sludge; SW = Surface Water; WW = Wastewater



4

800-227-4224  
Outside the United States, call 970-669-3050



# EPA APPROVED METHOD – DEFINITION

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# TNTplus – Test ‘N’ Tube Chemistries

## I) Vial and Reagents

DosiCap Zip containing stable freeze dried chemistry

Differentiation of ranges via Color coding

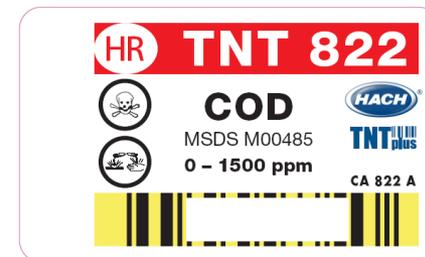
Strong brand recognition

Test name and (if so) safety information

Barcode for automatic test recognition

Exact amount of liquid reagent in vial

Freestanding 13mm vial due to flat bottom



Area for Sample ID

# TNTplus – Test ‘N’ Tube Chemistries

## II) Working Procedure in each box

Test, Parameter, Storage Conditions

Application range, safety information

Working Procedure w/ Pictogram

Principle and main interferences



**TNT 822**  
Chemical Oxygen Demand

0 – 1500 mg/L COD

Temperature of sample/reagent: 15 – 25°C



Storage



Protect against light



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

- **Range of application:** For water and wastewater; digestion is required
- Some of the chemicals and apparatus used in this procedure may be **hazardous to the health and safety of the user** if inappropriately handled or accidentally misused.
- Wear **appropriate eye protection and clothing** for adequate user protection. If contact occurs, flush the affected area with running water. Follow instructions carefully.
- Close the hood or place a **safety shield** in front of the COD reactor to prevent injury if splattering occurs.
- The reagent mixture is **light-sensitive**. Keep unused vials inside original closed box. Refrigerate if possible.
- Spilled reagent will affect **test accuracy** and is hazardous to skin and other materials. Wash spills with running water.

- 1 Preheat 150°C**  
Turn on the reactor. Preheat to 150°C. Close the hood or place the safety shield in front of the reactor.
- 2 2.0 mL Sample**  
Pipet carefully 2.0 mL of sample into the vial. Cap and clean the outside of the vial.
- 3 Invert!**  
Hold the vial by the cap over a sink. Invert **gently** several times to mix. The vial will become **very hot** during mixing. Place the vial into the preheated reactor.
- 4 Heat 150°C 2h**  
Heat the vial for **two hours**.
- 5 Wait! 20 min**  
Wait about **20 minutes** for the vial to cool to 120°C or less.
- 6 Invert carefully!**  
Invert the vial several times while **still warm**.
- 7 Cool before reading**  
Place the vial into a rack and cool to room temperature.
- 8 Read**  
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
The mg/L COD results are defined as the mg of O <sub>2</sub> consumed per liter of sample under conditions of this procedure. In this procedure, the sample is heated for two hours with a strong oxidizing agent, potassium dichromate. Oxidizable organic compounds react, reducing the dichromate ion to green chromic ion.	Chloride is the primary interference when determining COD concentration. Each COD vial contains mercuric sulfate that will eliminate chloride interference up to specified level (see table below).
TNT Test	Maximum Cl <sup>-</sup> concentration in sample (mg/L)
TNT 822	2000

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

# SL1000/PPA – PORTABLE WATER QUALITY TESTING



## 3 Steps to Faster, Highly Accurate Water Testing.

- 1 INSERT CHEMKEY™ REAGENTS
- 2 DIP INTO WATER SAMPLE
- 3 READ YOUR RESULTS

No zeroing, no mixing, no shaking,  
no chemicals or vials to handle.

# THE NEXT GENERATION OF FIELD TESTING - SL 1000 PORTABLE PARALLEL ANALYZER

## Test More Parameters in the Field in Less Time

Simultaneous measurement of up to 4 colorimetric and two electrochemistry parameters in under 8 minutes

## Eliminate Transcription Errors

Automatically records and stores measurement data along with sample site and time information

## Carry Less Equipment into the Field

One meter for photometry and electrochemistry, less paper, and organized in a portable workstation.



## Eliminate Testing Errors

- No Method Selection
- No Rinsing
- No Blank Preparation
- No Sample Volume
- No Zeroing
- No Mixing
- No Data Transcription
- No Lab Samples

Insert Chemkeys



Dip in Sample



Wait for Result



# TAKE HOME MESSAGE

- You are the first and most important step in the analytical process!
- Have a copy of your method/procedure.
- Use proper sampling and sample preservation techniques.
- Don't let temperature affect your results, or hurt your equipment.
- Change the batteries once a year!
- Use standards to check your equipment and your procedures. Verify your results!