-From Field to Laboratory-

The Basics of Sampling and Analysis Techniques for Wastewater Operators

William Romanelli, MS & Kristen Thomas, MPA

-Field Science Technician- -Laboratory Production Coordinator-

Water Pollution Control Laboratory, Bureau of Environmental Services
City of Portland, OR

Field Operations Section,

Environmental Investigations Division

-Monitoring & Sampling-

- Surface Water
- Groundwater
- Stormwater
- Industrial Wastewater
- Collection system flow monitoring
- Watershed Health Assessments
- Soil, Sediment, Ditchings
- Green Infrastructure Performance (Green Streets & Ecoroofs)
- Temperature
- Fish Tissue
- Methane

9 Full-Time Staff, Plus Supervisor

WPCL Laboratory,

Environmental Investigations Division

-Sample Analysis-

- Lab Manager
- 14 Lab Staff
  - 6 Analysts
  - 5 Specialists
  - 3 Coordinators

- 7 days/week, 365 days/year
- Wastewater is our specialty!
  - Inorganics
  - Organics
  - Micro

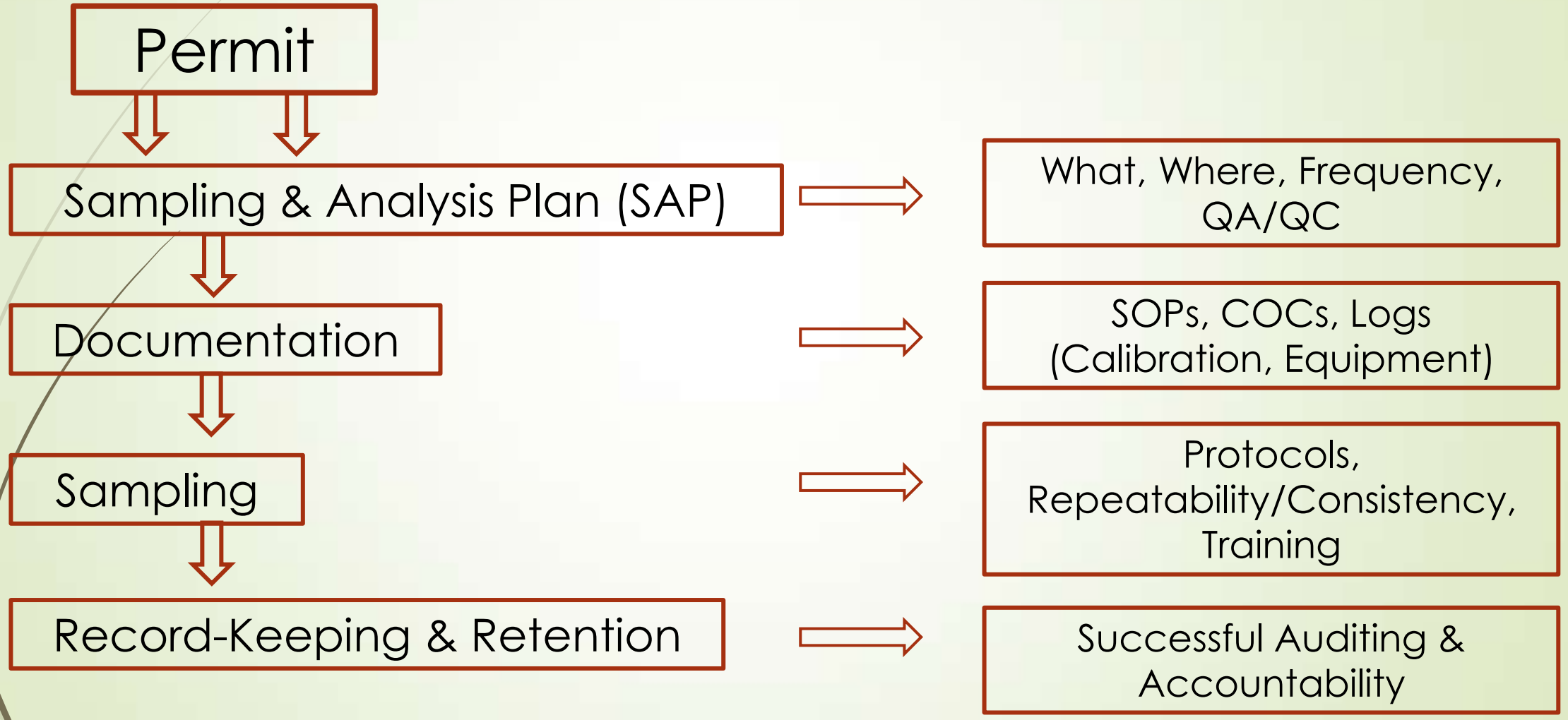
Overview

- Your NPDES Permit & the Ensuing Process
- Sampling & Analysis Plan
- Documentation & Accountability
- Selective Sampling & Ethics
- Quality Assurance/Quality Control
- Documentation & Record-Keeping
- Representative Collection of Analytes

Your NPDES Permit

- Schedule A – discharge limits
- Schedule B – monitoring/reporting requirements
  - Sampling location
  - Frequency of Sampling (seasonal?)
  - Type of sample (grab, comp, measurement)
  - Reporting Requirements/Protocols
    - DLs/QLs
  - Pretreatment, biosolids, WET
  - Tier 1 Monitoring (mets, orgs)
- Analysis – Methods, QA/QC
  - 40 CFR 136

The Process of Implementation

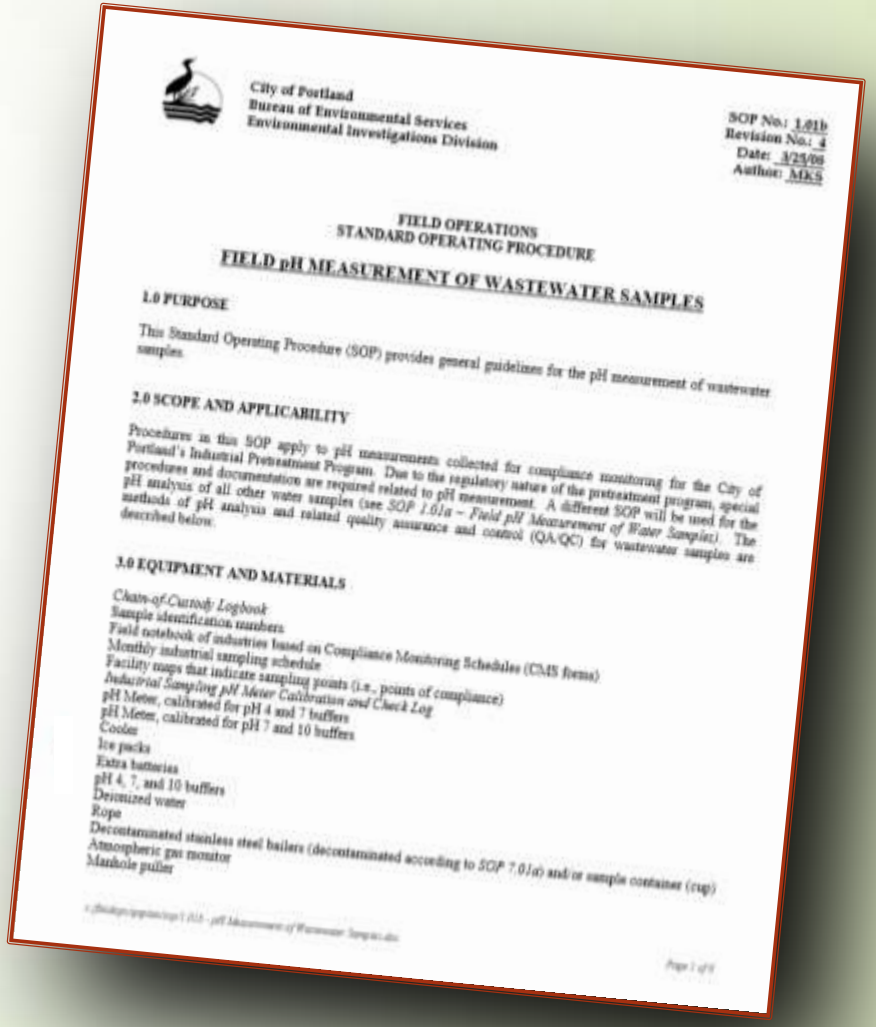


Documentation

- Storage & Access
  - Permit
  - Sampling & Analysis Plan (SAP)
  - Source Materials
  - Communications
  - Standard Operating Procedures (SOPs)
  - Chains of Custody (COCs)
  - Logs
    - Equipment Calibration (Daily Check-Out/Check-In)
    - Equipment Repair/Replacement

Documentation

- Standard Operating Procedure (SOP)
  - Purpose
  - Scope & Applicability
  - Equipment & Materials
  - Procedure: Step-By-Step
  - Potential Problems
  - QA/QC
  - Site Safety & Access
  - Resources/References



City of Portland
Bureau of Environmental Services
Environmental Investigations Division

SOP No.: 1.01b
Revision No.: 4
Date: 3/25/08
Author: MKS

FIELD OPERATIONS
STANDARD OPERATING PROCEDURE

FIELD pH MEASUREMENT OF WASTEWATER SAMPLES

1.0 PURPOSE

This Standard Operating Procedure (SOP) provides general guidelines for the pH measurement of wastewater samples.

2.0 SCOPE AND APPLICABILITY

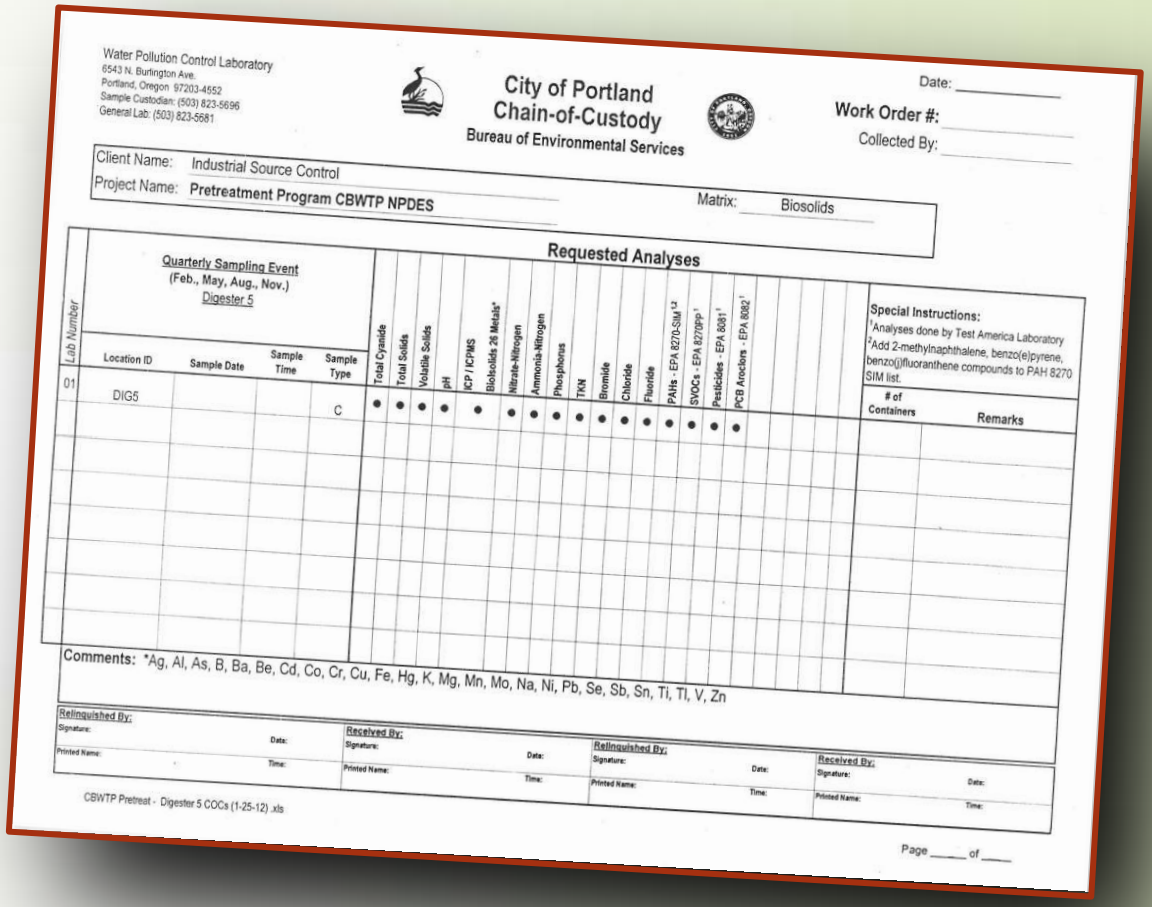
Procedures in this SOP apply to pH measurements collected for compliance monitoring for the City of Portland's Industrial Pretreatment Program. Due to the regulatory nature of the pretreatment program, special procedures and documentation are required related to pH measurement. A different SOP will be used for the pH analysis of all other water samples (see *SOP 1.01a – Field pH Measurement of Water Samples*). The methods of pH analysis and related quality assurance and control (QA/QC) for wastewater samples are described below.

3.0 EQUIPMENT AND MATERIALS

Chain-of-Custody Logbook
Sample identification numbers
Field notebook of industries based on Compliance Monitoring Schedules (CMS forms)
Monthly industrial sampling schedule
Facility maps that indicate sampling points (i.e., points of compliance)
Industrial Sampling pH Meter Calibration and Check Log
pH Meter, calibrated for pH 4 and 7 buffers
pH Meter, calibrated for pH 7 and 10 buffers
Cooler
Ice packs
Extra batteries
pH 4, 7, and 10 buffers
Deionized water
Rope
Decontaminated stainless steel bailers (decontaminated according to *SOP 7.01a*) and/or sample container (cup)
Atmospheric gas monitor
Manhole puller

Documentation

- COC
  - Possession
  - Sampling Personnel ID
  - Organization ID
  - Analytes Requested
  - Project ID & Details
  - Time & Date of Collection
  - Sampling Location
  - Specific Instructions to Lab



Water Pollution Control Laboratory
6543 N. Burlington Ave.
Portland, Oregon 97203-4552
Sample Custodian: (503) 823-5696
General Lab: (503) 823-5681

City of Portland
Chain-of-Custody
Bureau of Environmental Services

Date: _____
Work Order #: _____
Collected By: _____

Client Name: Industrial Source Control
Project Name: Pretreatment Program CBWTP NPDES
Matrix: Biosolids

Quarterly Sampling Event
(Feb., May, Aug., Nov.)
Digester 5

Requested Analyses

Lab Number	Location ID	Sample Date	Sample Time	Sample Type	Total Cyanide	Total Solids	Volatile Solids	pH	ICP / ICPMS Biosolids 26 Metals*	Nitrate-Nitrogen	Ammonia-Nitrogen	Phosphorus	TKN	Bromide	Chloride	Fluoride	PAHs - EPA 8270-SIM [1,2]	SVOCs - EPA 8270Pp [1]	Pesticides - EPA 8081 [1]	PCB Aroclors - EPA 8082 [1]	# of Containers	Remarks
01	DIG5			C	●	●	●	●	●	●	●	●	●	●	●	●	●	●	●	●		

Special Instructions:
[1]Analyses done by Test America Laboratory
[2]Add 2-methylnaphthalene, benzo(e)pyrene, benzo(j)fluoranthene compounds to PAH 8270 SIM list.

Comments: *Ag, Al, As, B, Ba, Be, Cd, Co, Cr, Cu, Fe, Hg, K, Mg, Mn, Mo, Na, Ni, Pb, Se, Sb, Sn, Ti, Tl, V, Zn

Relinquished By:	Received By:	Relinquished By:	Received By:
Signature:	Signature:	Signature:	Signature:
Printed Name:	Printed Name:	Printed Name:	Printed Name:
Date:	Date:	Date:	Date:
Time:	Time:	Time:	Time:

CBWTP Pretreat - Digester 5 COCs (1-25-12) .xls

Page ____ of ____

Date: _____

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City of Portland Chain-of-Custody

Bureau of Environmental Services

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01	DIG5			C	●	●	●	●	●	●	●	●	●	●	●	●	●	●	●	●

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Comments: *Ag, Al, As, B, Ba, Be, Cd, Co, Cr, Cu, Fe, Hg, K, Mg, Mn, Mo, Na, Ni, Pb, Se, Sb, Sn, Ti, Tl, V, Zn

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Page _____ of _____

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01	DIG5			C	●	●	●	●	●	●	●	●	●	●	●	●	●	●	●	●		

Special Instructions:
[1]Analyses done by Test America Laboratory
[2]Add 2-methylnaphthalene, benzo(e)pyrene, benzo(j)fluoranthene compounds to PAH 8270 SIM list.

Comments: *Ag, Al, As, B, Ba, Be, Cd, Co, Cr, Cu, Fe, Hg, K, Mg, Mn, Mo, Na, Ni, Pb, Se, Sb, Sn, Ti, Tl, V, Zn

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Signature: Date:	Signature: Date:	Signature: Date:	Signature: Date:
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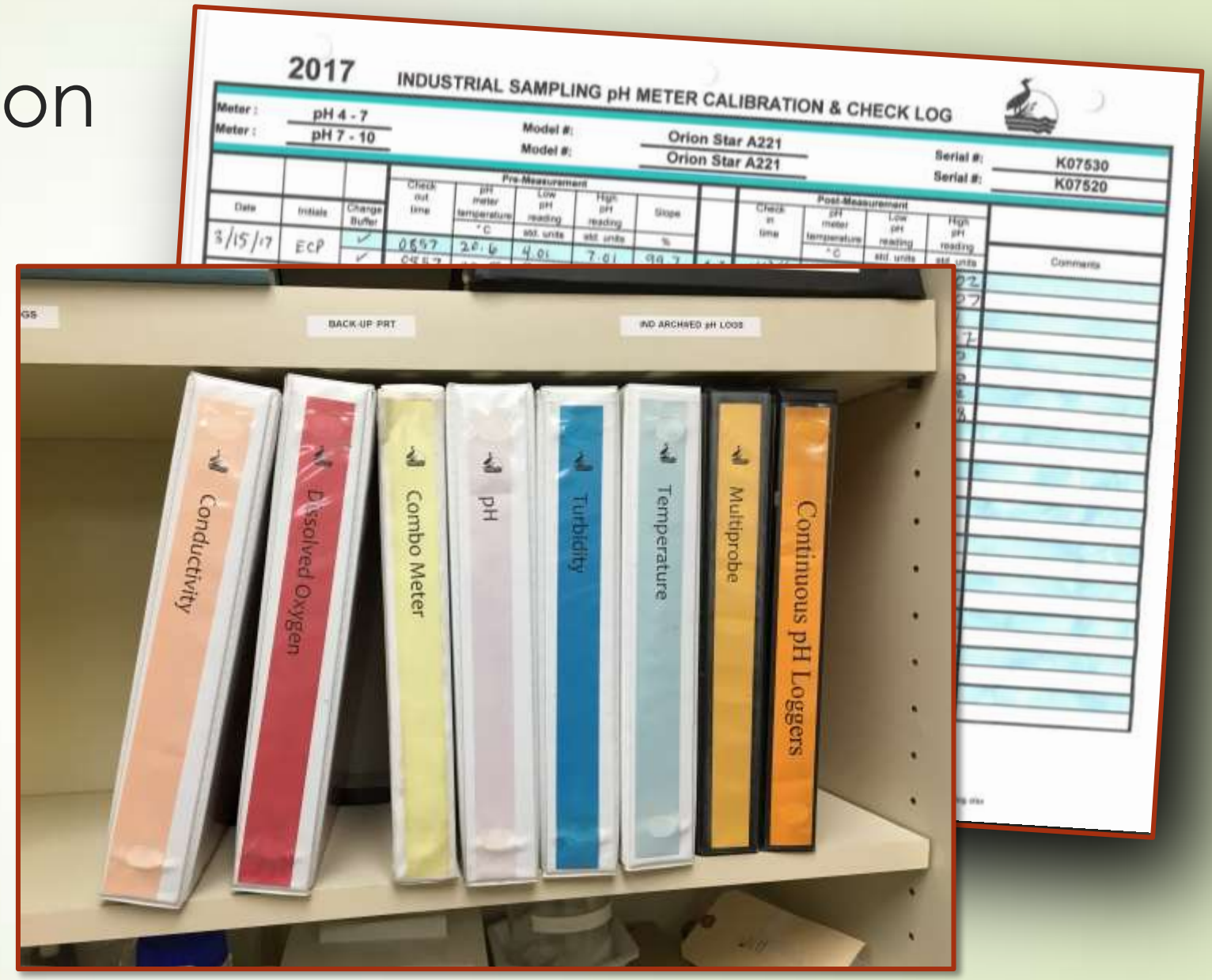
Documentation

- Logs
  - Calibration
  - Equipment

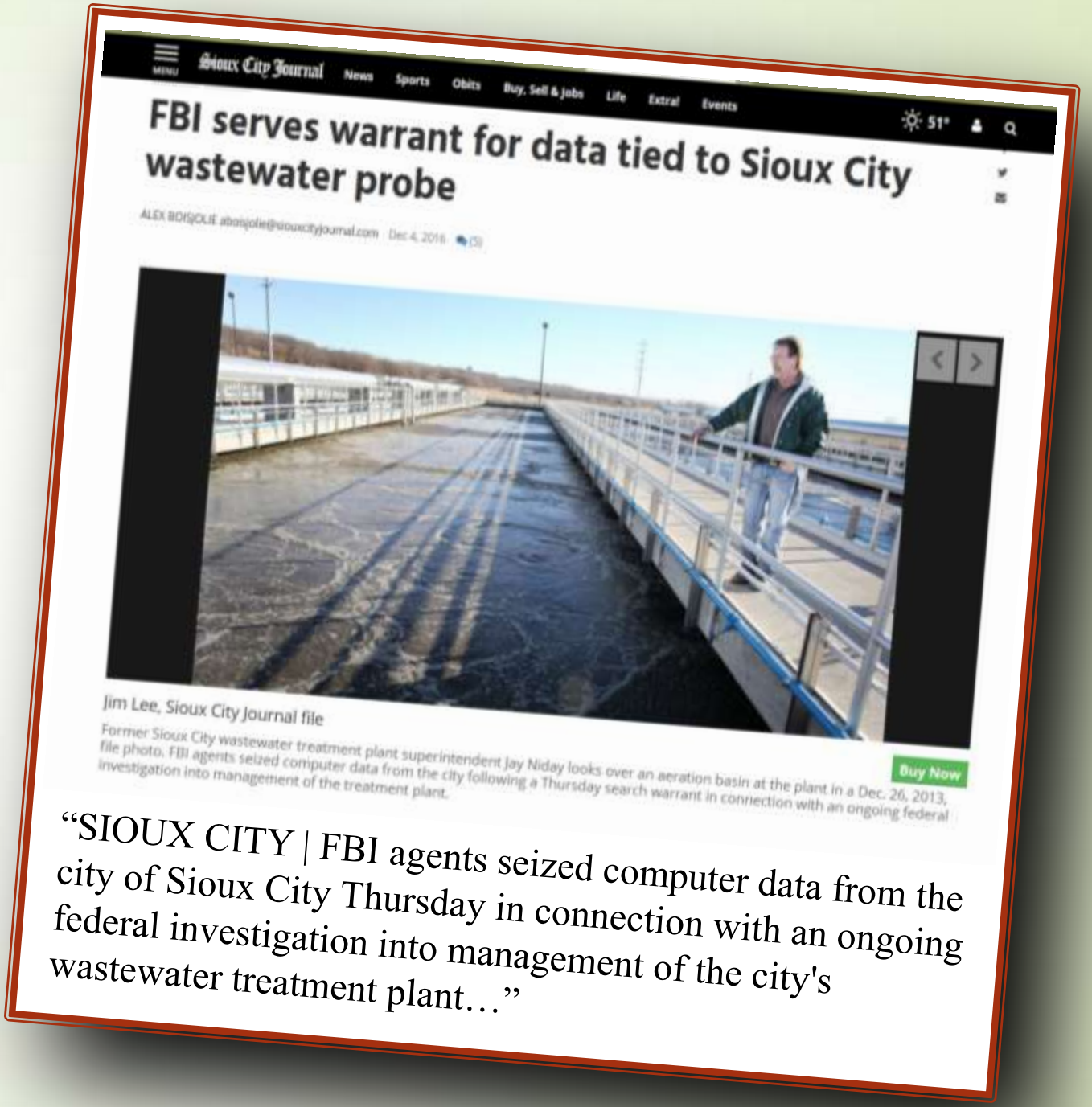


Documentation & Accountability

"If you didn't write it down,

you didn't do it."

Selective Sampling



Sioux City Journal

MENU News Sports Obits Buy, Sell & Jobs Life Extra! Events 51°

FBI serves warrant for data tied to Sioux City wastewater probe

ALEX BOISJOLIE aboisjolie@siouxcityjournal.com Dec 4, 2016 (3)

Jim Lee, Sioux City Journal file

Former Sioux City wastewater treatment plant superintendent Jay Niday looks over an aeration basin at the plant in a Dec. 26, 2013, file photo. FBI agents seized computer data from the city following a Thursday search warrant in connection with an ongoing federal investigation into management of the treatment plant.

Buy Now

"SIOUX CITY | FBI agents seized computer data from the city of Sioux City Thursday in connection with an ongoing federal investigation into management of the city's wastewater treatment plant…"

Selective Sampling

- "…plant supervisors Niday and Schwarte had dramatically raised chlorine and bisulfate doses on days that E. coli samples were taken and then reduced the levels….at least four other city employees took part in the manipulation of test results on directions from Niday and Schwarte…"
- "…which resulted in…discharges into the Missouri River to contain high levels of E.coli bacteria, potentially endangering public health…"
- The Missouri River is known as a recreational river.
- The practice dated back to 2011, approximately 5 years.
- "…the city saved at least $100,000 in one year when workers administered the smaller levels of chlorine…"
- "…Schwarte and Niday were both dismissed by the city, and they agreed to surrender their state wastewater licenses…"
- "…the U.S. Attorney is considering criminal charges related to the case…"

Quality Assurance/Quality Control (QA/QC)

- QA: A system of prevention of mistakes and defects.
- QC: A system of detection of mistakes and defects.

Glove Choices

- Nitrile: A good general purpose glove.
- Latex: Contains metals (Zn, Ni, Cu) contamination on the surface.
- Vinyl: Not for organics sampling.

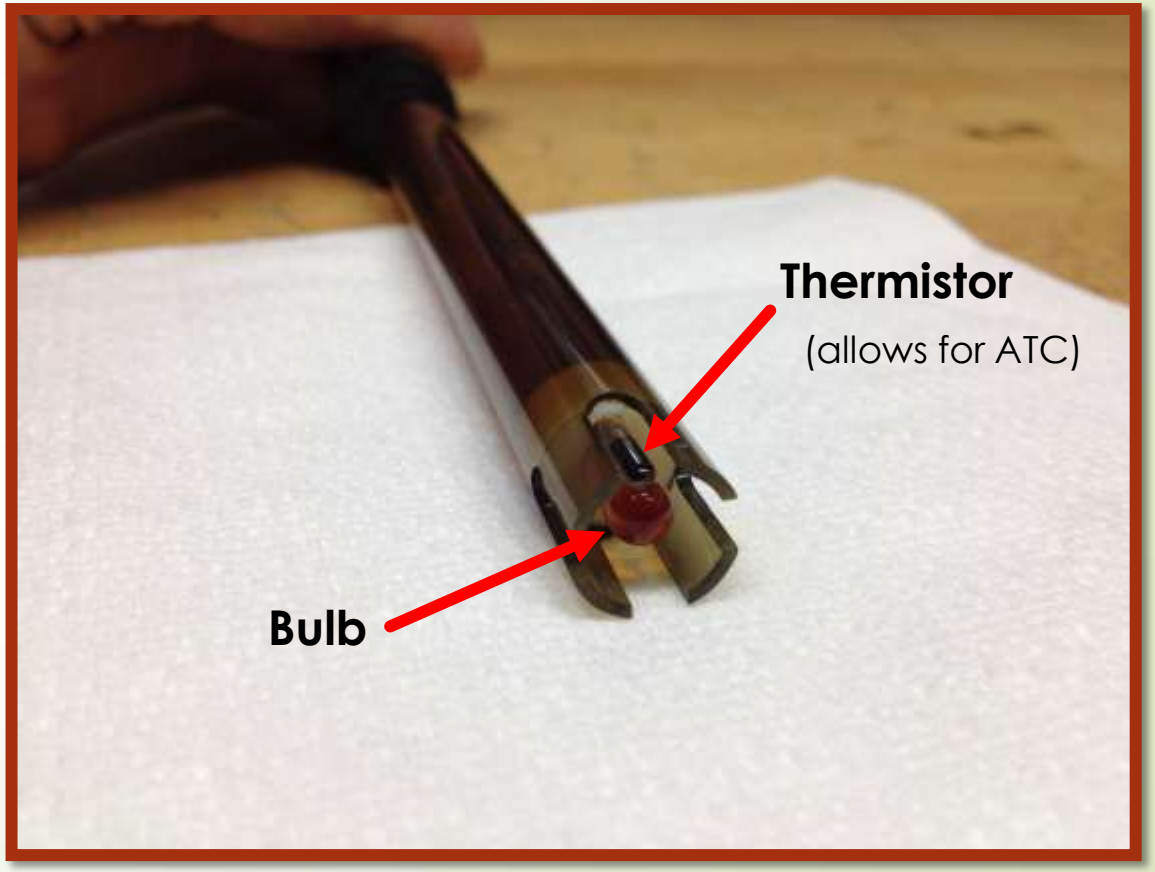
Contamination from Gloves

- Drips: from your gloves (or anything else)
- Latex Gloves: Carry metals on their surfaces (Zn, Ni, Cu).
- Vinyl Gloves: Not for organics sampling. ("Like" does not like "Like")
- Powdered Gloves: Simply a bad idea.

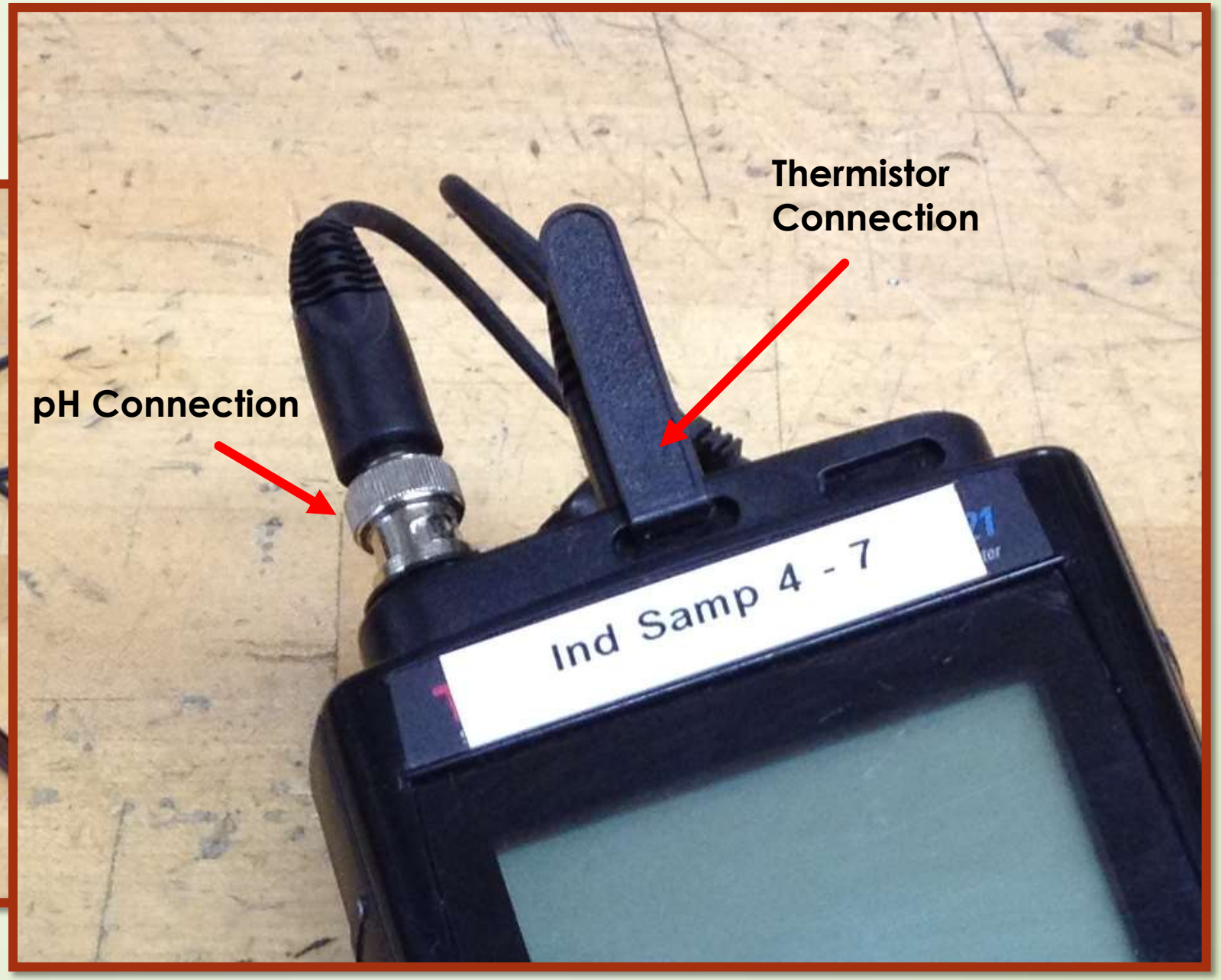
pH (more than a number)

- Quality of instrument
- Automatic Temperature Compensation (ATC)
- Proper calibration
- Quality of buffers
- Life Expectancy of Probes
- Drift & !Shock!
- Extreme Weather

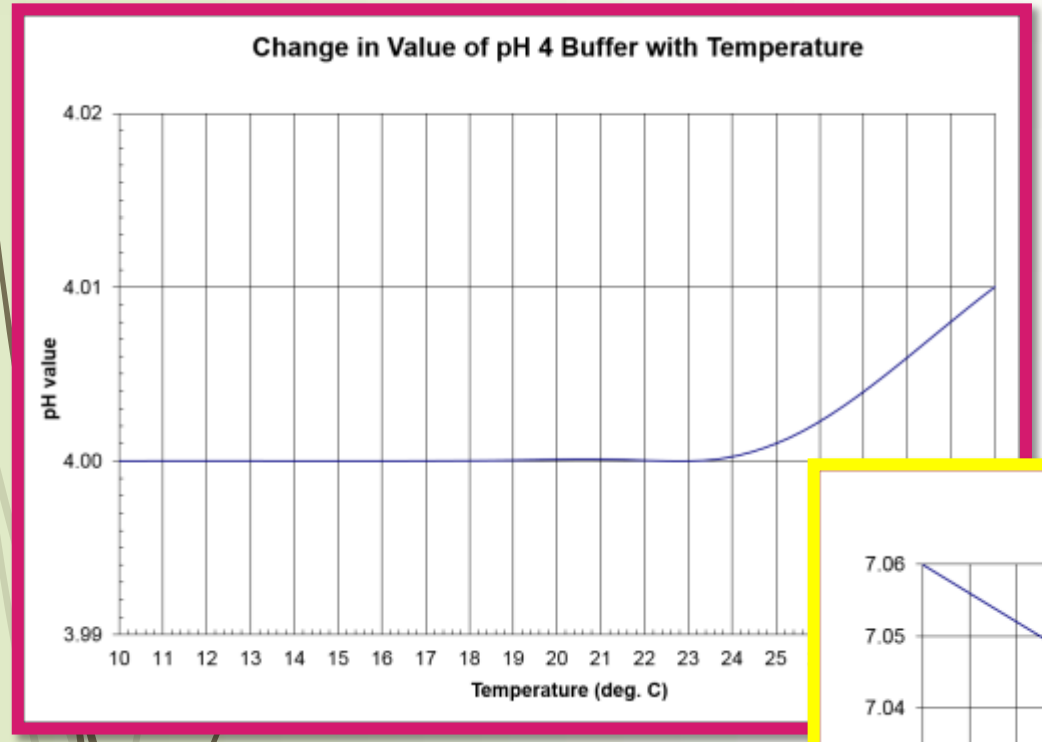
Get a Quality Instrument

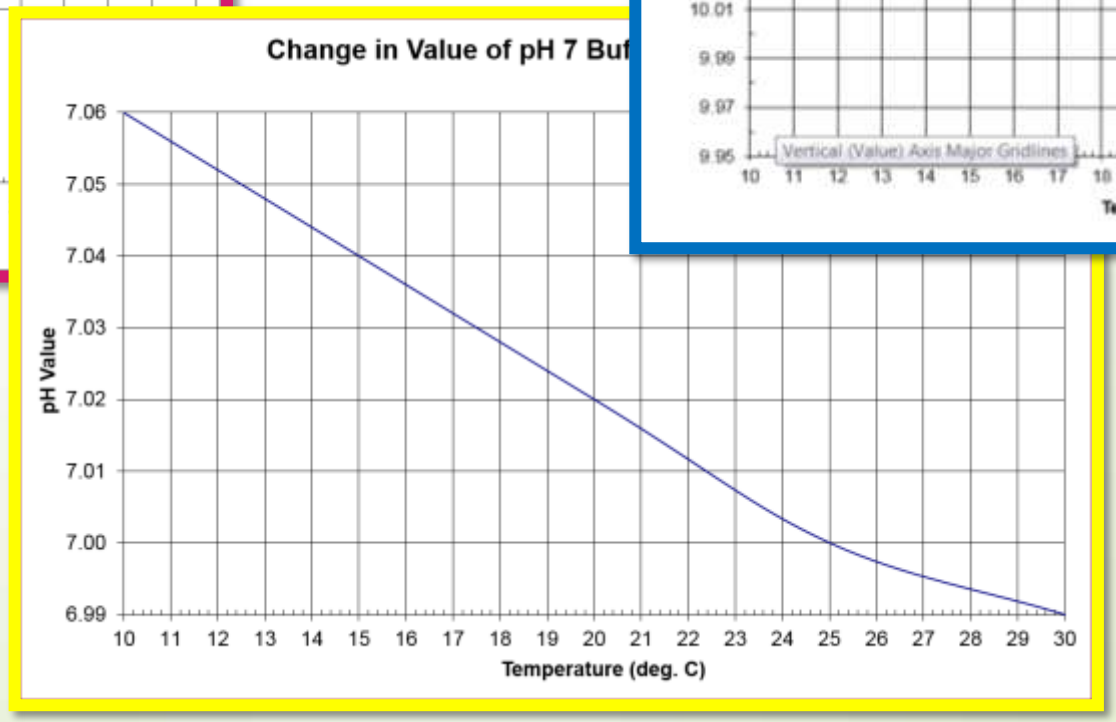


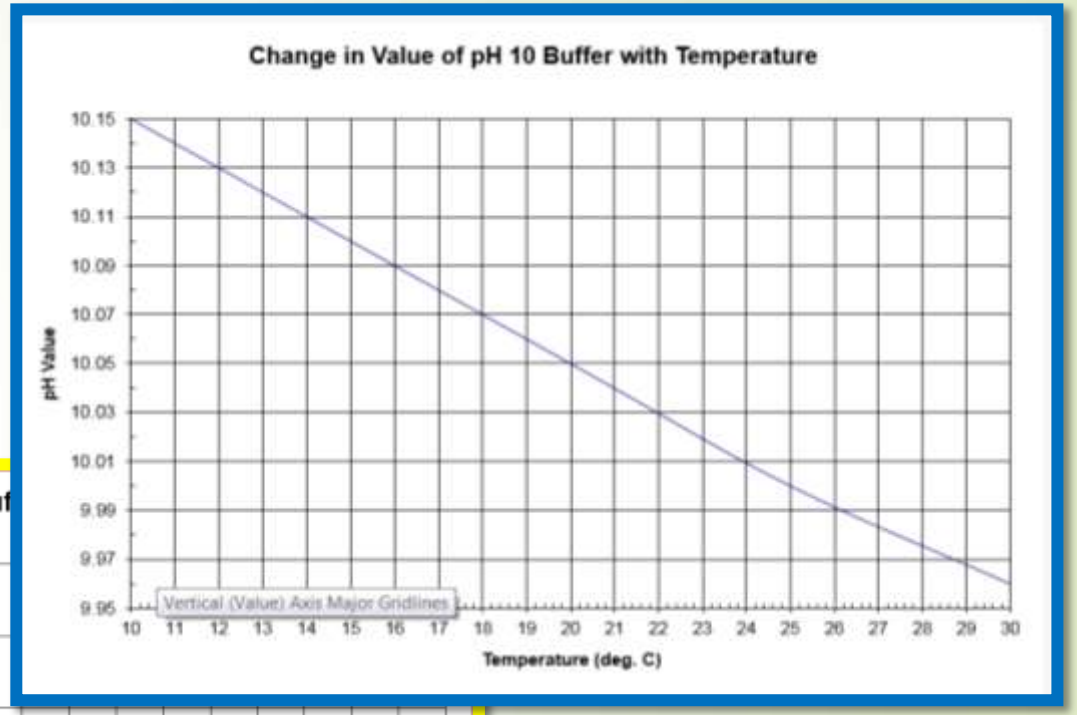
Quirks: Meter



Automatic Temperature Compensation (ATC)



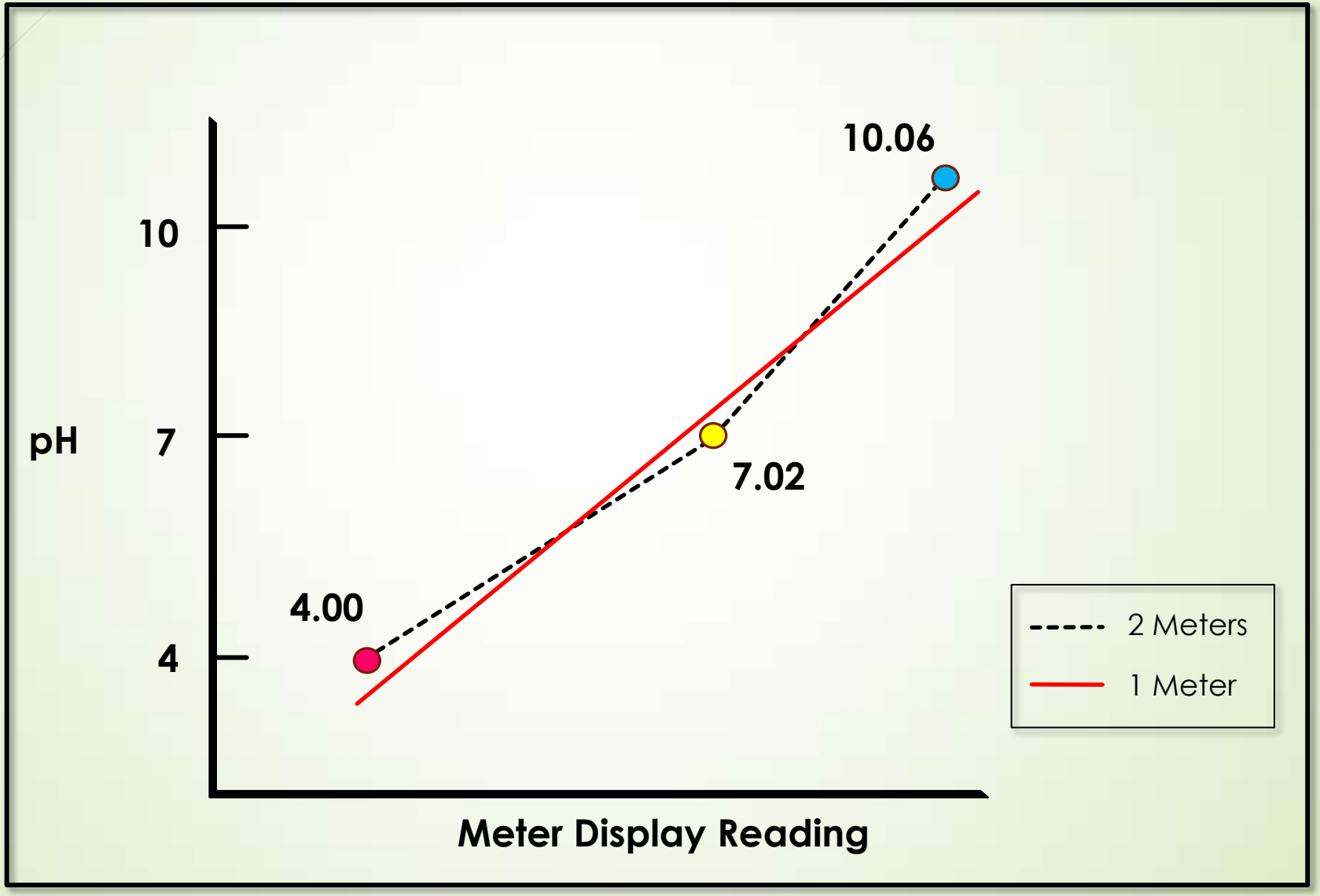




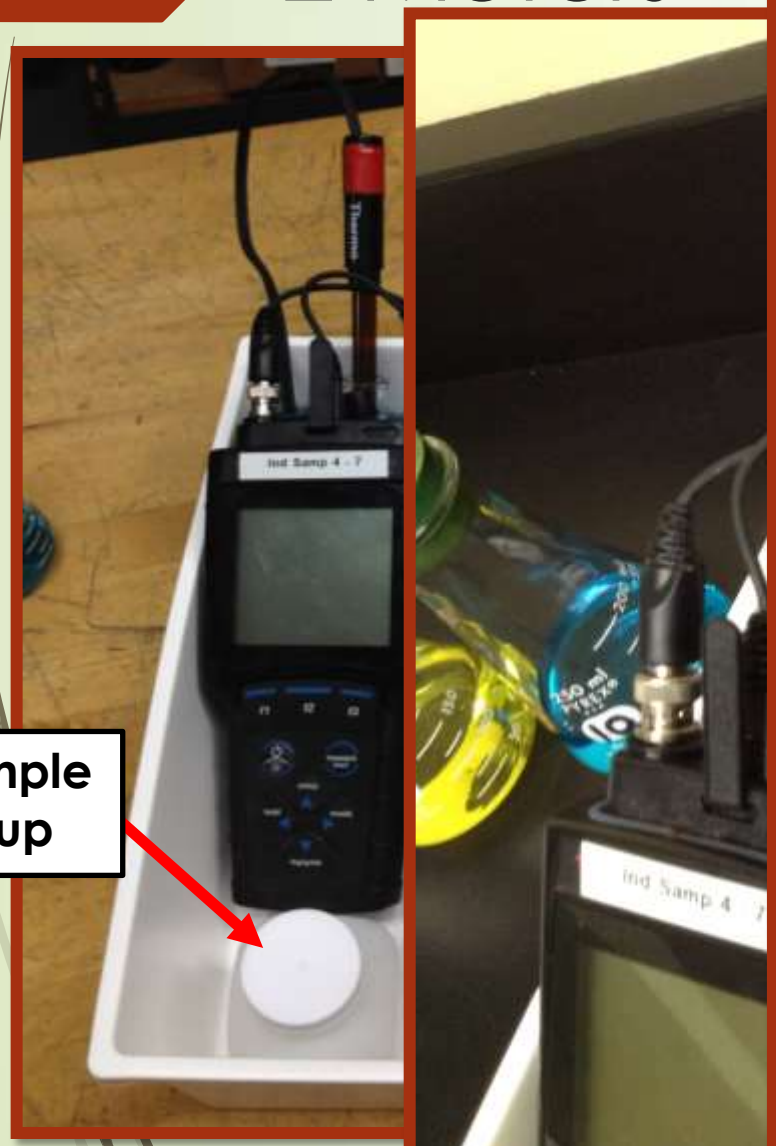
2 Meters

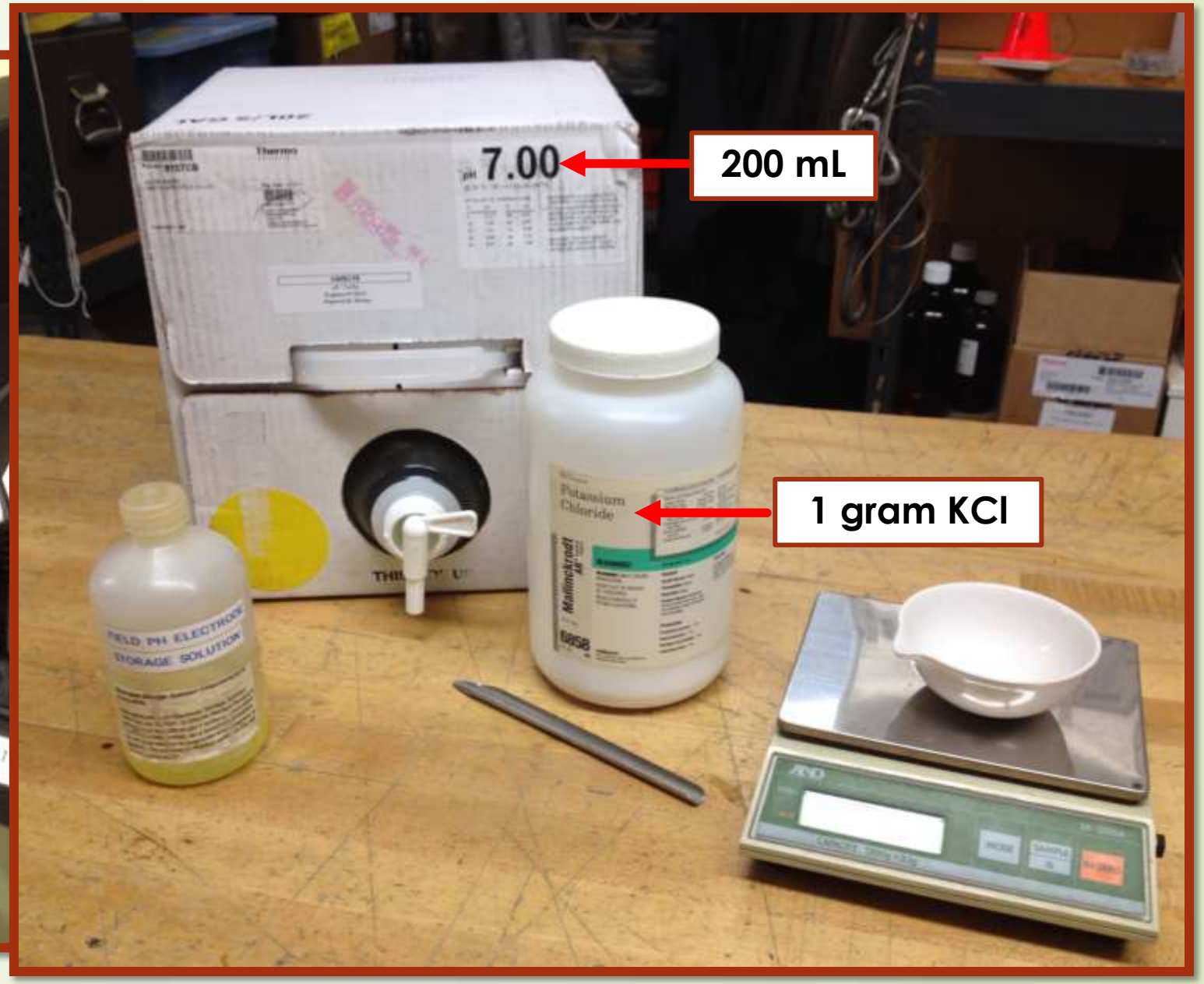
One Meter or Two ?

(pH values of buffers at 19° C)



2 Meters





Sample Cup

Calibration Documentation

INDUSTRIAL SAMPLING pH METER CALIBRATION & CHECK LOG

Meter : pH 4 - 7 | Model #: Orion 3 Star | Serial #: 007172

			Pre-Measurement						Post-Measurement			
			Check out time	pH meter temperature	Low pH reading	High pH reading	Slope		Check in time	pH meter temperature	Low pH reading	High pH reading
Date	Initials	Change Buffer		°C	std. units	std. units	%			°C	std. units	std. units
		4 - 7										
		7-10										
		4 - 7										
		7-10										
		4 - 7										
		7-10										
		4 - 7										
		7-10										
		4 - 7										
		7-10										
		4 - 7										
		7-10										

Post-Measurement acceptable range: 3.90 - 4.10, 6.90 - 7.10, and 9.90 - 10.10 at 25C (*see graphs for range at other temperatures*)

If meter checks in outside of the acceptance range: **A)** If there were **no violations** that day, simply note in comments that meter failed check-in.

Calibration Documentation

INDUSTRIAL SAMPLING pH METER CALIBRATION & CHECK LOG

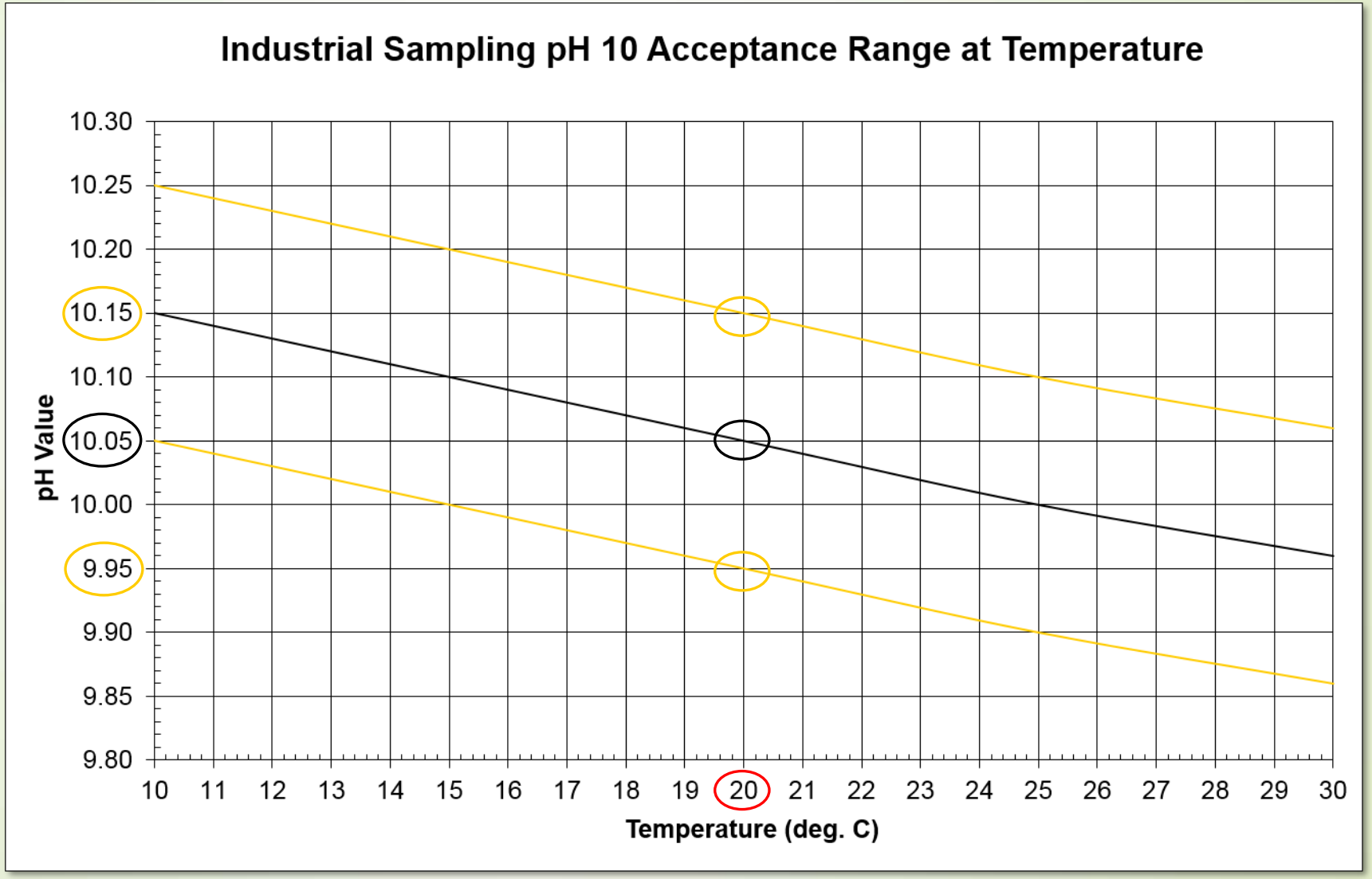
Meter :	pH 4 - 7		Model #:	Orion 3 Star	Serial #:	007172
Meter :	pH 7 - 10		Model #:	Orion 3 Star	Serial #:	007342

			Pre-Measurement						Post-Measurement				
Date	Initials	Change Buffer	Check out time	pH meter temperature	Low pH reading	High pH reading	Slope		Check in time	pH meter temperature	Low pH reading	High pH reading	Comments
								4 - 7					
								7-10					
								4 - 7					
								7-10					
								4 - 7					
								7-10					
								4 - 7					
								7-10					
								4 - 7					
								7-10					
								4 - 7					
								7-10					

Post-Measurement acceptable rang[illegible] 3.90 - 4.10, 6.90 - 7.10, and 9.90 - 10.10 at 25C (*see graphs for range at other temperatures*)

If meter checks in outside of the acceptan[illegible]nge: **A)** If there were **no vio[illegible]tions** that day, simply note in comments that meter failed check-in.

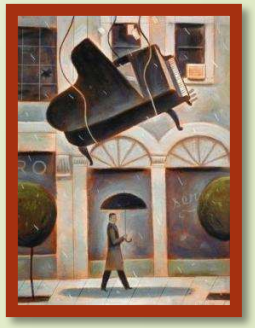
Check-In/Check-Out Tolerances



More pH Caveats

Meters:

- Shelf-Life of Probes (6-18 Months)
- Temperature Extremes in Field
- Gel-Filled vs Oil-filled Probes (temperature equilibration)
- Extreme pHs can cause drift in calibration upon check-in.



More pH Caveats

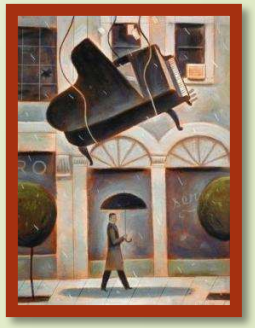
Buffers:

- Freshness Counts, on a Daily Basis
- Exposure to Air
- Expiration Dates
- Beware of Buffer 10?
- Best Stored at Room Temperature

More pH Caveats

Sample Integrity:

- Clean container
- Instantaneous read is best.
- “Hold” of 15 minutes, Maximum!
- Not Glove-Sensitive



Cyanide

- Grab or Composite
- Pre-Preserved sample container (NaOH)
- Keep containers separated
- Hold is 14 Days, beginning at end of composite.
- Chill sample

Residual Chlorine

- Grab Sample or Continuous Measurement
- Hold of 15 minutes – ASAP!
- No Preservation
  - Keep it Cold
  - Keep it Dark

E. coli

- Grab
- Sterile 250mL bottle, Sealed
- Leave Headspace
- Cap & Chill Immediately
- Hold 8 hours
- Contamination from air, splashes, sprays…

Oil and Grease (OG)

- Grab
- Glass only
- Collect directly in sample bottle, no transfer
- Hold is 28 Days
- Chill
- Lab-preserved with HCl

Biological Oxygen Demand (BOD)

- Grab or Composite
- Chilled composite sampler
- Hold is 48 Hours, begins at end of composite
- Chill at all times

Total Suspended Solids (TSS)

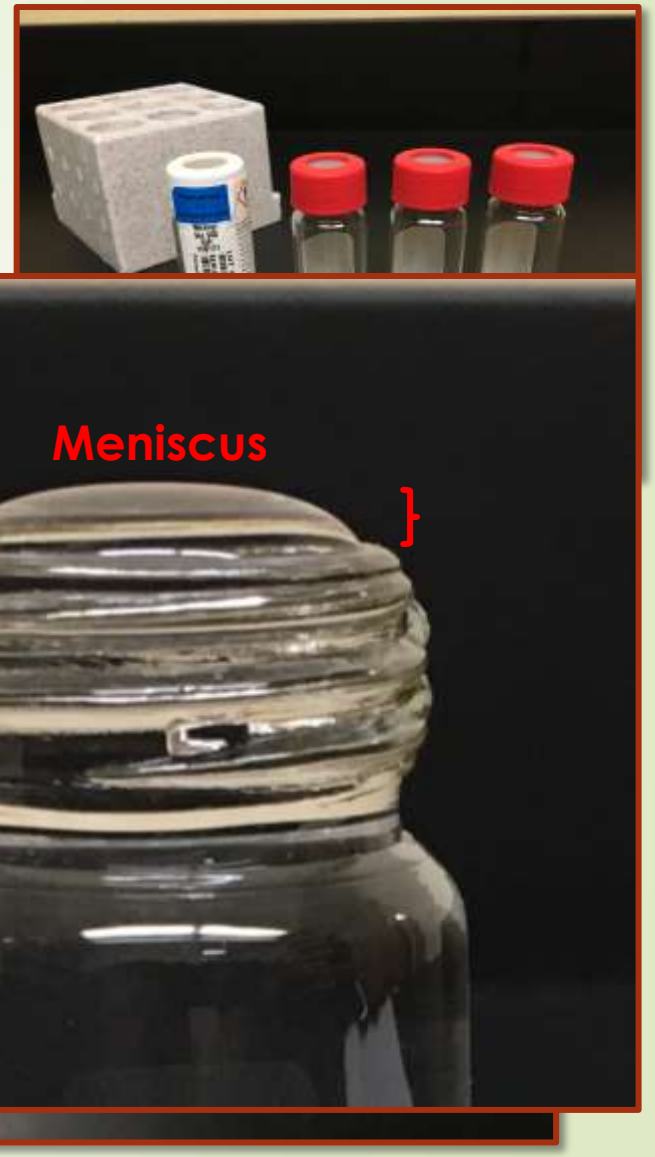
- Grab or Composite
- Chilled Composite Sampler
- Hold is 7 Days
- Chill at all times

Metals

- Grab or Composite
- Pint for analysis
- No powdered or latex gloves!
- Hold is180 Days, but Hg is 28.
- Chilling not required
- Preserved in lab with Nitric HNO3

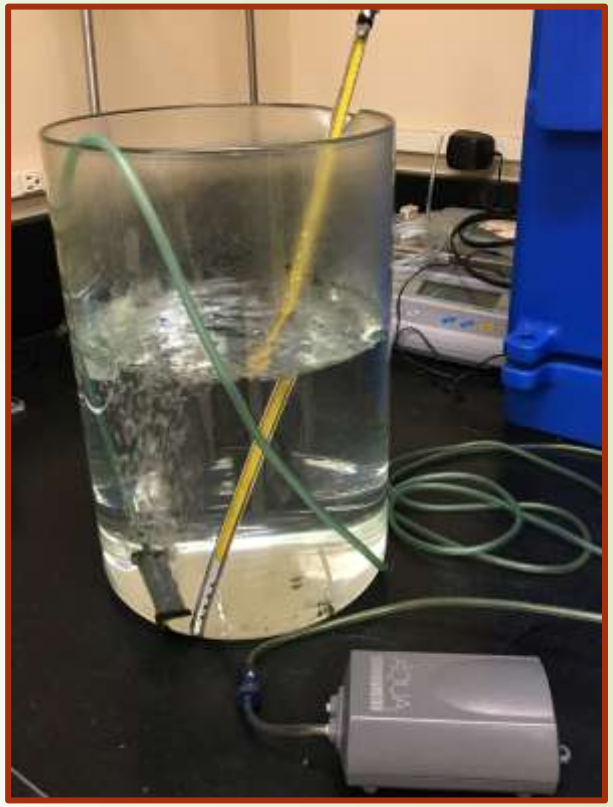
Volatile Organic Carbon (VOC)

- Grab
- 40 mL Vials: Preserved (HCl) and/or Non-Preserved
- Hold is variable
- Zero “Headspace”
- Blank: Ultra-Pure De-Ionized Water (DI)
- Never write on/near septum
- Chill



Temperature

- Immediate read
- Check In/Out Log
- Reference Thermometer in water bath circulated 24/7
- NIST = National Institute of Standards and Technology
- Periodic official calibration check on Reference



Questions…

Abbey Mills Pump Station, England

Bureau of Environmental Services,
City of Portland