

# What happens to the Samples after collection?

Just what DOES the lab do with  
samples after they are collected?

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# Explanation of sample types



# Grab

- A single, one time “grab” of sample



# Grab

- analysis of pH, temperature, cyanide, total phenols, residual chlorine, oil and grease, fecal coliform (including *E. coli*), and *Enterococci* (previously known as fecal streptococcus), or volatile organics, grab samples must be collected for those pollutants. 40 CFR Part 136

# Composite

- a 24-hour composite sample, using a minimum of four (4) grab samples, must be used unless specified otherwise at 40 CFR Part 136

# Composite

- Multiple samples grabbed, then placed together into the same container

# Youngstown sampler

ISCO external



ISCO internal



# Struthers sampler

**Sigma**



**Sigma controller**







**Carboys**

➤ Characteristics and sampling frequency are determined by the NPDES permit



➤ Common characteristics include:

- CBOD
- TSS
- NH<sub>3</sub>
- Total Phosphorous
- Temperature
- pH
- DO

## ➤ Characteristics we monitor/test for:

- Temperature, DO, TSS, Oil & Grease
- Nitrogen- Ammonia, Nitrate-nitrite, TKN
- Total Phosphorous, Hexavalent Chromium
- Free Cyanide, Chloroform, CBOD
- Flow rate, Fecal coliforms, Mercury

## ➤ Characteristics we monitor/test for:

- Total residual chlorine
- Metals, total recoverable: Nickel, Silver, Zinc, Cadmium, Lead, Chromium, Copper
- Bioassay: Acute & Chronic toxicity – both fat-head minnows and water fleas

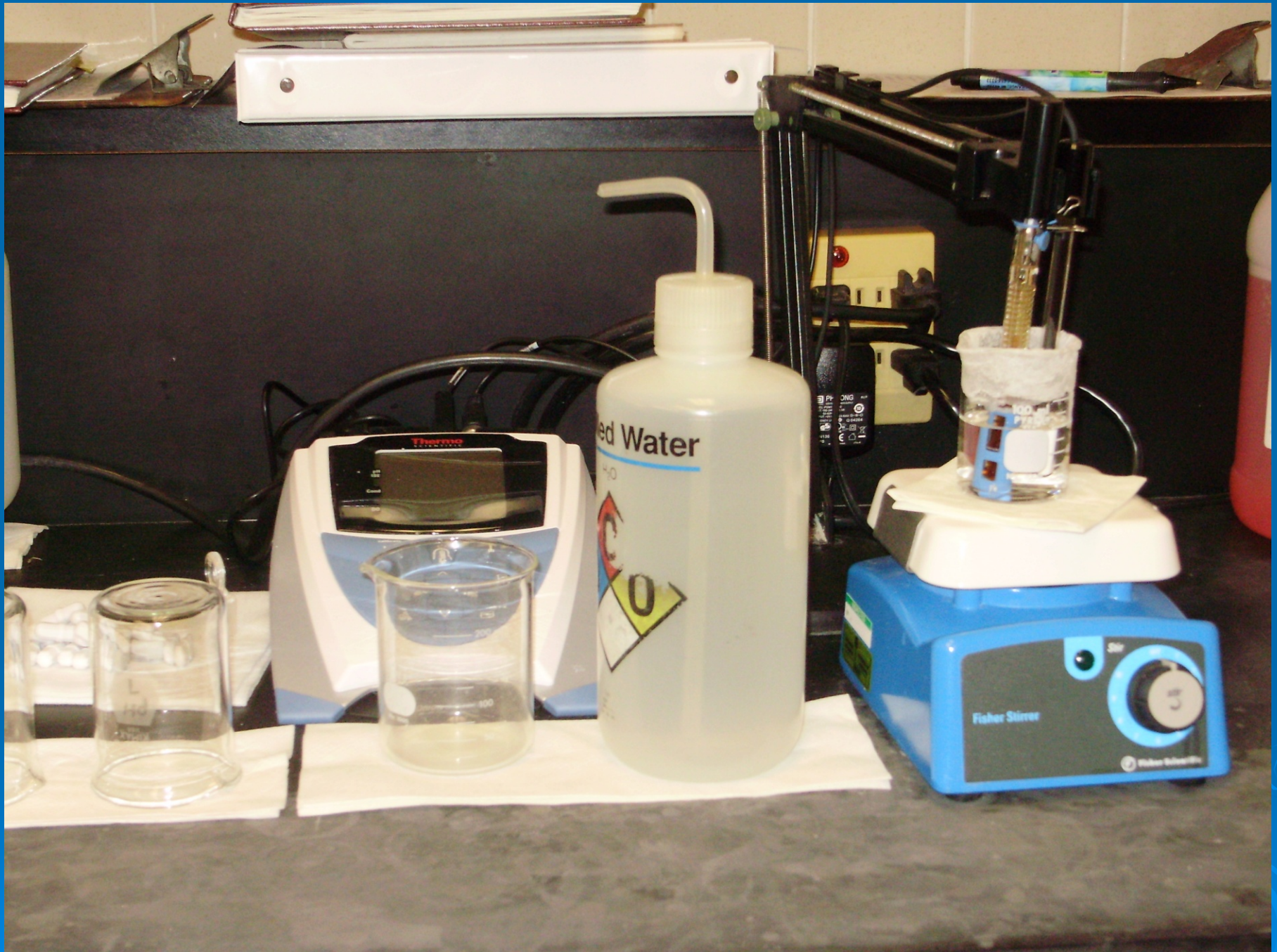
# Grab Samples



- Daily pH, temperature and dissolved oxygen (D.O.) check to ensure the automatic system stays in calibration for both the raw and final.
- Grab samples, analyzed within 15 minutes of collection/ delivery to lab

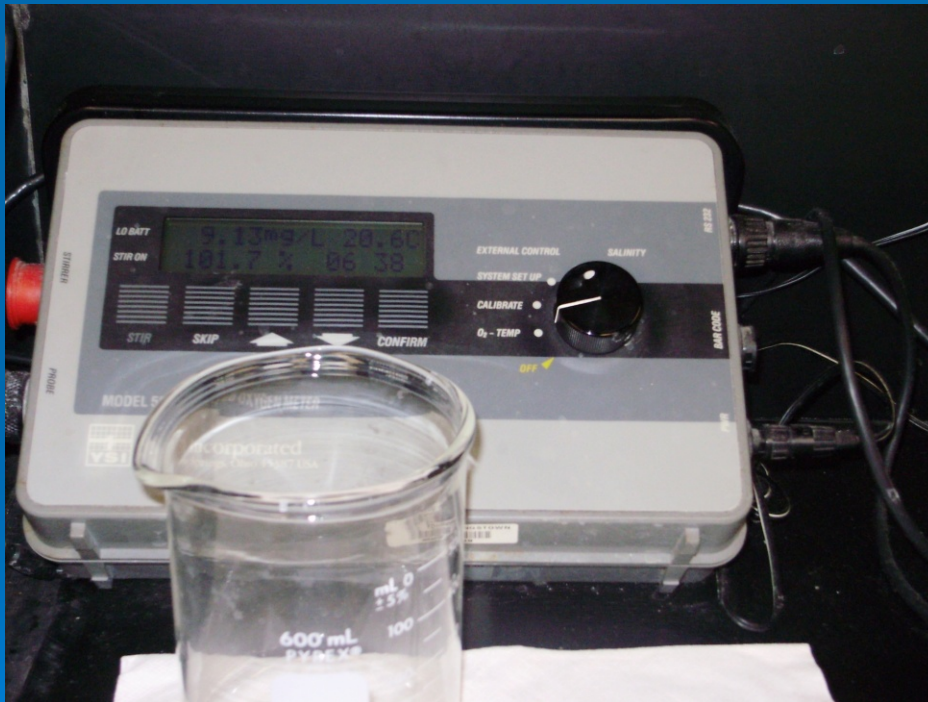
- pH and temperature are checked on a Thermo scientific 5 star meter/ probe



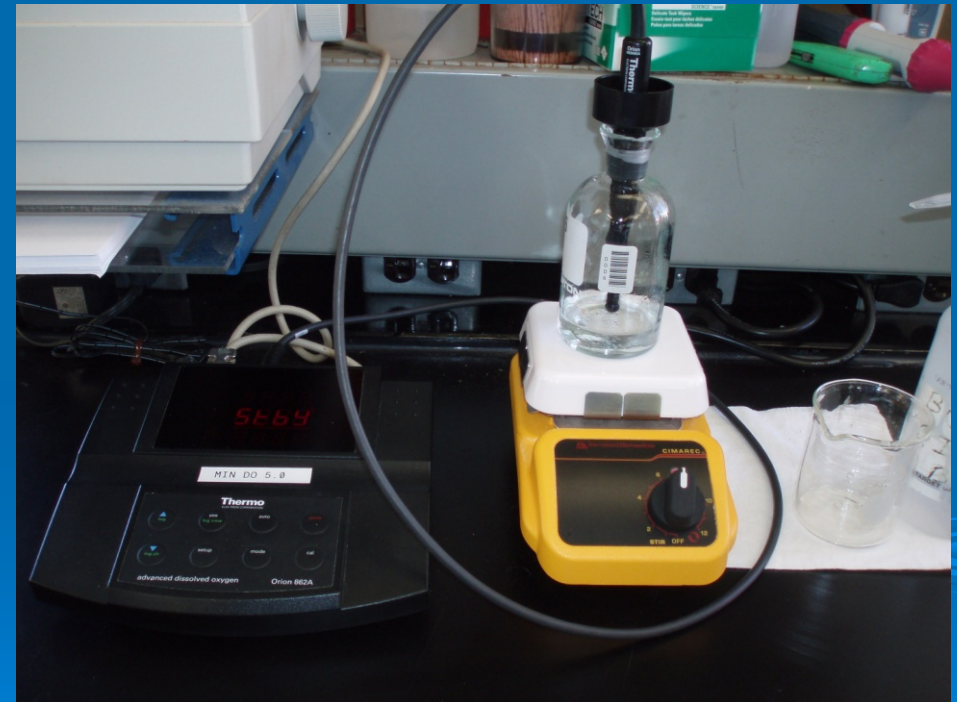


- D.O. is checked on a YSI meter/ probe
- Also commonly used are Thermo-Orion meter/ probe combo

## Youngstown: YSI system



## Geneva: Thermo-Orion system



- Chlorine residual is tested/  
monitored 3 times daily during the  
summer months to report a  
maximum discharge  
concentration

- Chlorine concentration is also checked once daily at the base of the cascade, and again after the contact tanks to ensure adequate chlorine is being applied for disinfection.

- Chlorine residual is checked by amperometric titration
- Permit limit is 0.024 mg/L, ML is 0.050 mg/L according to the EPA. Violations do not occur until the ML has been exceeded.
- Grab samples, analyzed within 15 minutes of collection/ delivery to lab



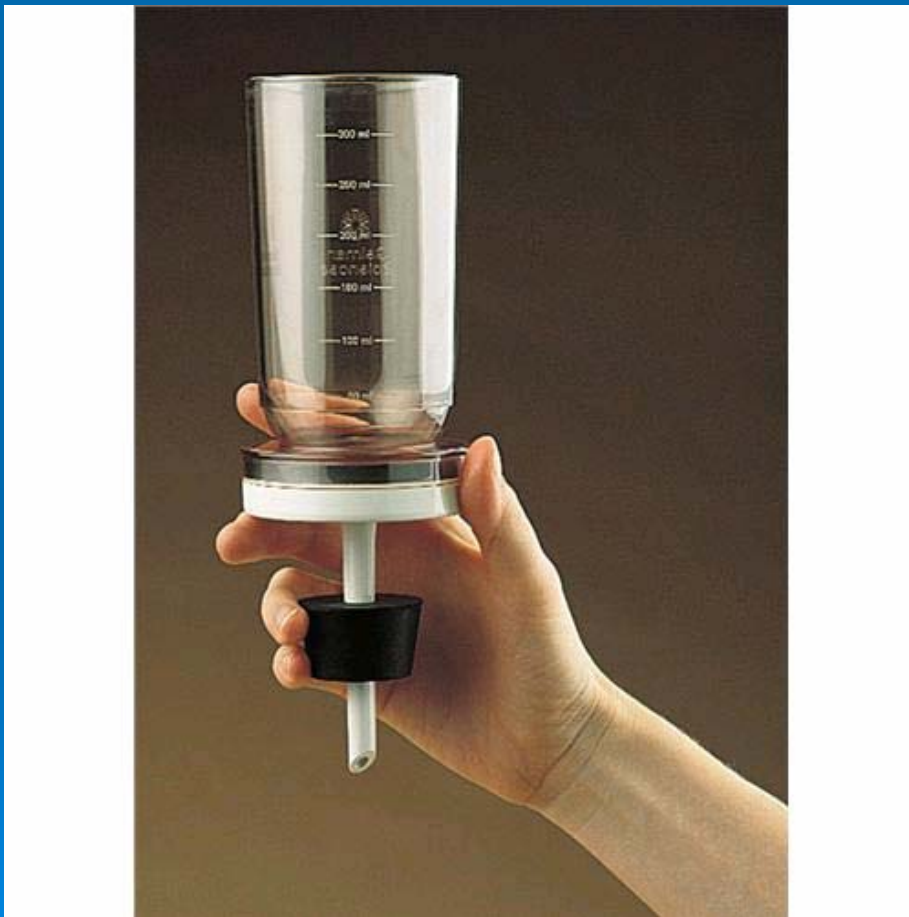
- Fecal coliforms, total coliforms and E. coli are daily tests during the summer months
- Samples must be analyzed within 6 hours of collection.
- Samples are incubated for 24 hours before reading.





# Filtering set

## Magnetic funnel

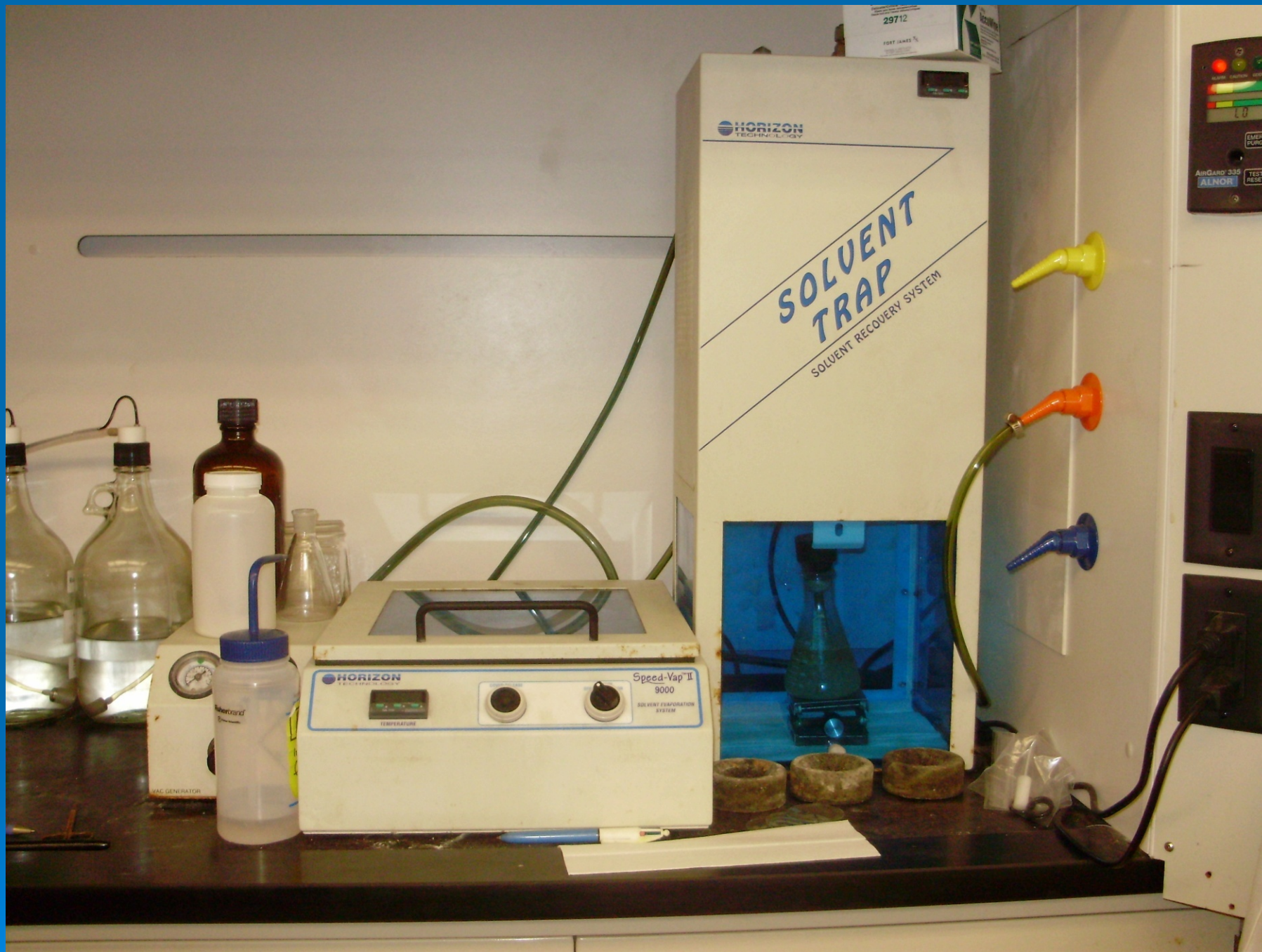


## Glass funnel



- Oil & Grease samples are grab samples, but can be preserved with acid to a pH <2 and stored for up to 28 days.
- We do Oil & grease by EPA method 1664A, automated solid phase extraction with n-hexane
- Can also be done by liquid to liquid extraction







YOUNGSTOWN WWTP LABORATORY  
SAMPLE ID: CA010-001 PRESERVATIVE  
COLLECTION DATE: 9-9-08  
LOG NUMBER: 08-211  
TEST REQUEST: O + G, A

YOUNGSTOWN WWTP LABORATORY  
SAMPLE ID: MA004-001 PRESERVATIVE  
COLLECTION DATE: 9-8-08  
LOG NUMBER: 08-207  
TEST REQUEST: O + G, A

YOUNGSTOWN WWTP LABORATORY  
SAMPLE ID: MA002-001 PRESERVATIVE  
COLLECTION DATE: 9-10-08  
LOG NUMBER: 08-213  
TEST REQUEST: O + G, A

YOUNGSTOWN WWTP LABORATORY  
SAMPLE ID: MA002-004 PRESERVATIVE  
COLLECTION DATE: 9-11-08  
LOG NUMBER: 08-214  
TEST REQUEST: O + G, C

YOUNGSTOWN WWTP LABORATORY  
SAMPLE ID: CA005-001 PRESERVATIVE  
COLLECTION DATE: 9-15-08  
LOG NUMBER: 08-218  
TEST REQUEST: O + G, B

# Composite

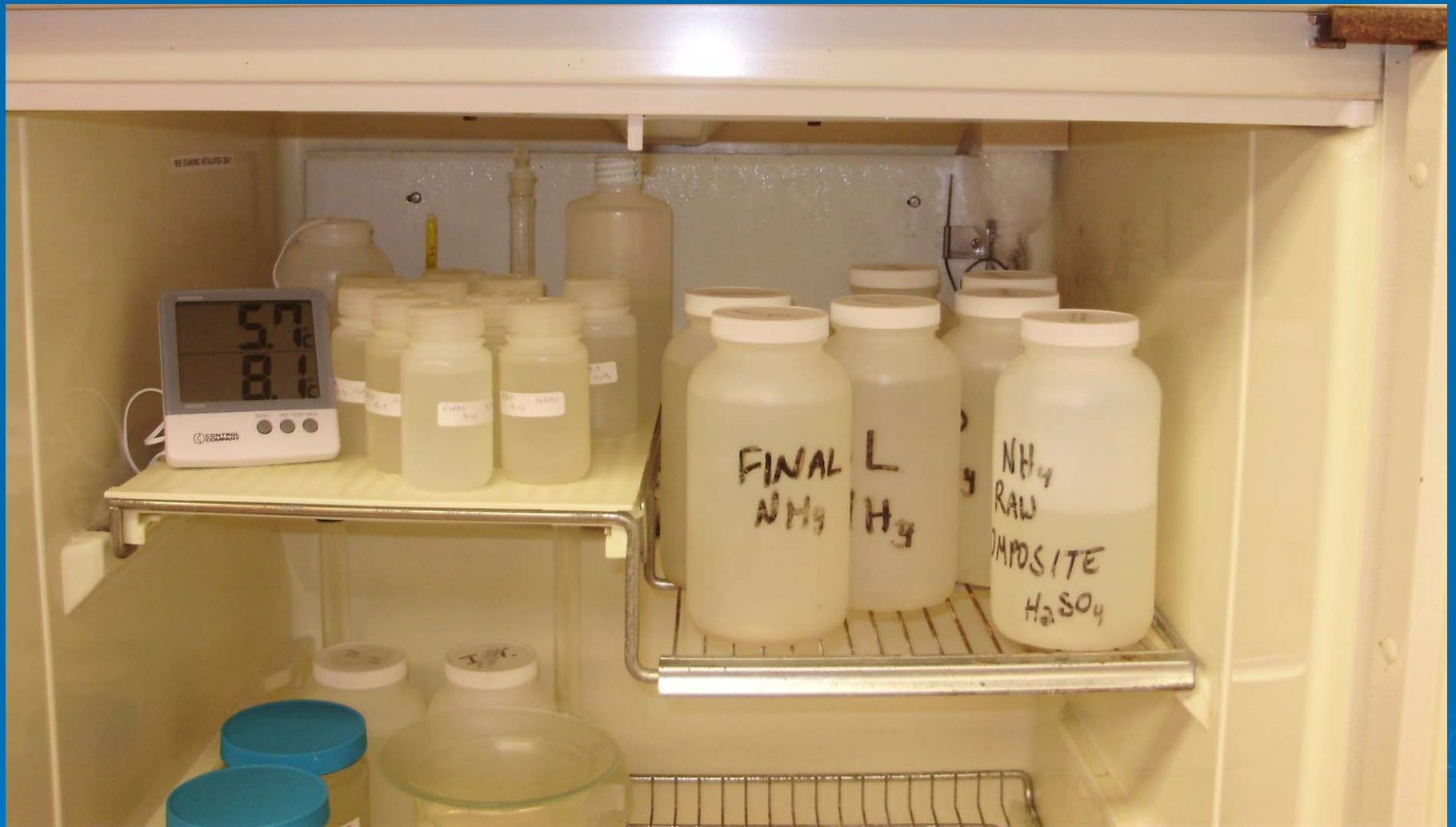


- Composite samples are brought into the lab, where they are split out according to the requirements for the day's analysis.



seed is 300ml in a filled (DI) 1 liter  
Blank and Raw are not seeded  
50ml Control = 2 shots







- Total suspended solids analysis are performed daily
- Samples can be held, unpreserved, for up to 28 days at  $\leq 6^{\circ}\text{C}$
- Samples are analyzed in accordance with SM 2540-D.



- CBOD's are collected daily.
- Samples can be held, unpreserved, for up to 48 hours after collection at  $\leq 6^{\circ}\text{C}$
- Sample sets are put on for analysis on Monday, Wednesday and Friday.
- There is a 5 day incubation period

# BOD incubators



- Metals samples: Cd, Cr, Cu, Pb, Ni, Zn, Ag for raw and final are collected once every 2 weeks.
- Samples are preserved with acid to a pH <2 and can be held for up to 6 months.
- Samples are digested for total recoverable metals in a programmable hot block, in accordance with EPA method 200.7



# Hot block digesters

**Youngstown**



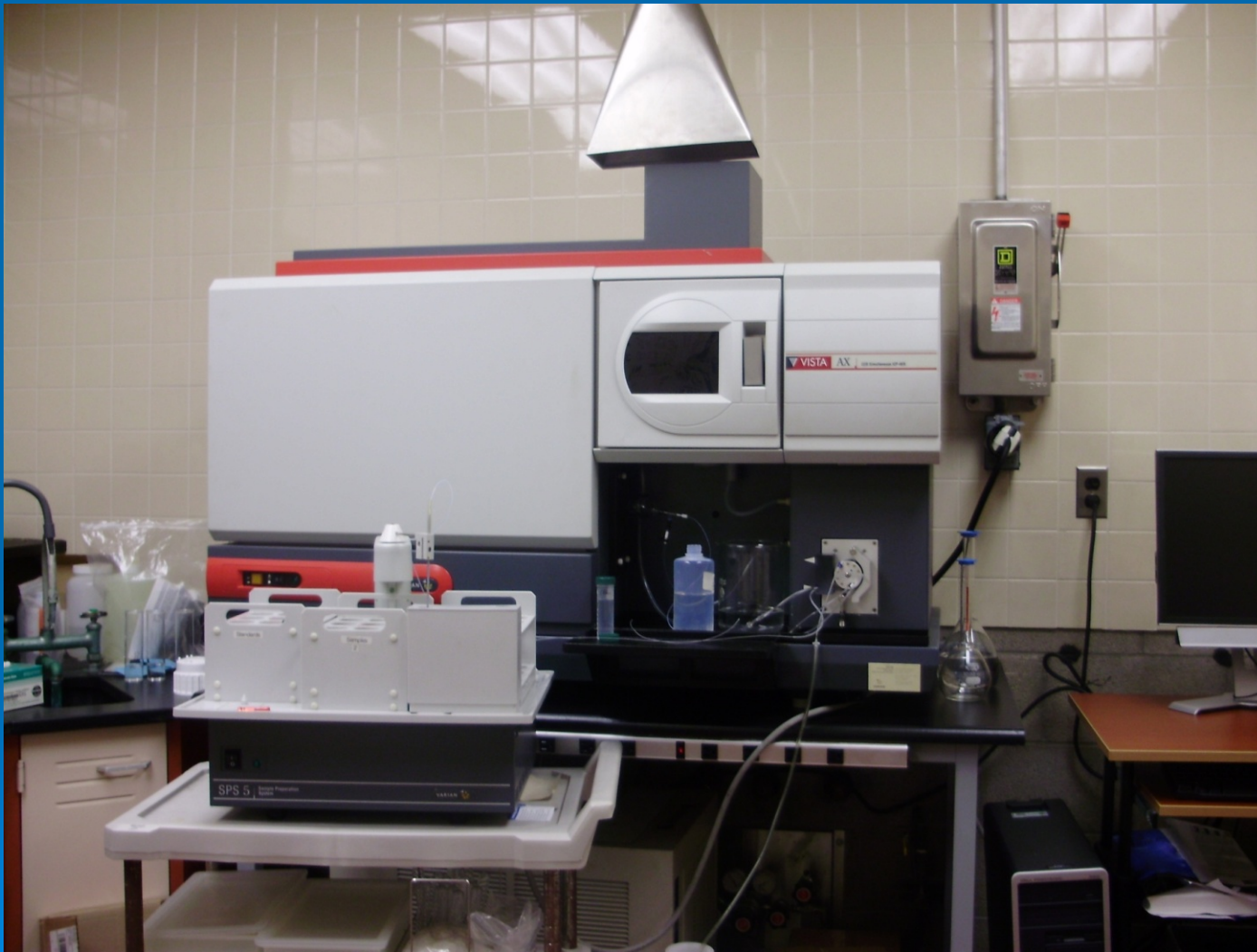
**Geneva**



- Sludge metals, composited, are prepped in the hot block, but the digestion step includes heating with a 30% hydrogen peroxide solution to break any organic bonds
- Sludge metals are collected once a month, by compositing over an 8 to 16 hour period
- Sludge is analyzed for As, Be, Cd, Cr, Cu, Pb, Ni, Mo, Se, Zn

- Prepped, digested metals samples are analyzed by ICP, in accordance with EPA method 200.7
- Metals can also be analyzed by AA

# ICP



# AA

**Struthers Flame AA**



**Struthers Furnace AA**



- Phenols (industrial and background samples only)
- Batch analysis, including a digested blank, standard, duplicate and spike
- Analyzed on the Westco Smartchem



# Smartchem Spec





# Specs

## Struthers



## Geneva



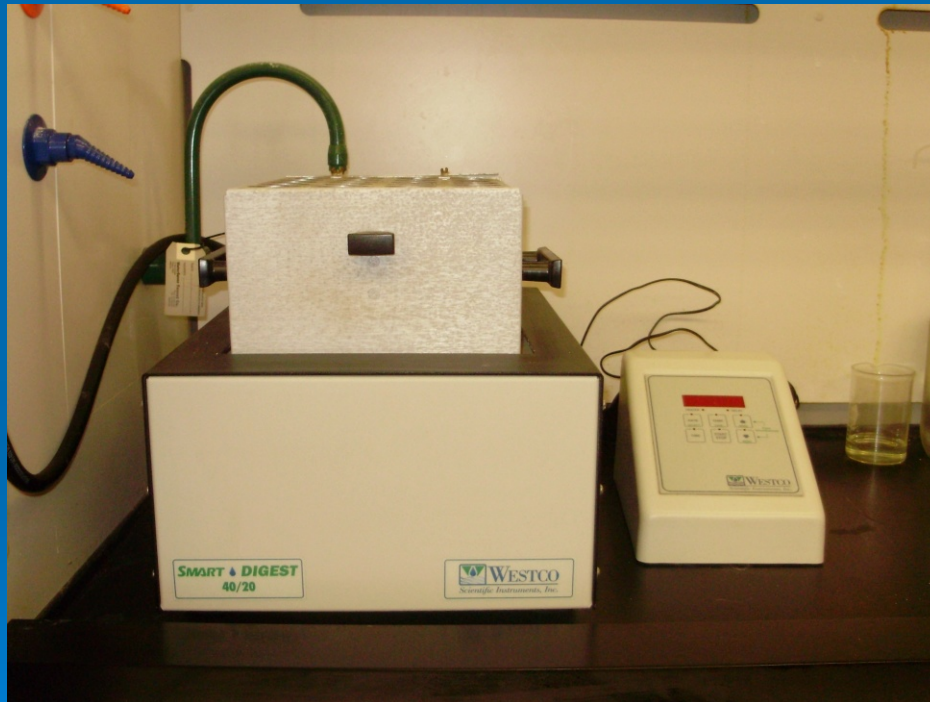
➤ Also analyzed on the Westco Smartchem:

- Hexavalent Chromium
- Nitrate-nitrite
- TKN
- Total Phosphorous

- TKN and total phosphorous digested on Westco Smartdigest (in Youngstown)
- Other digestion apparatus for TKN are hot block system

# TKN digestion

**Smart block**



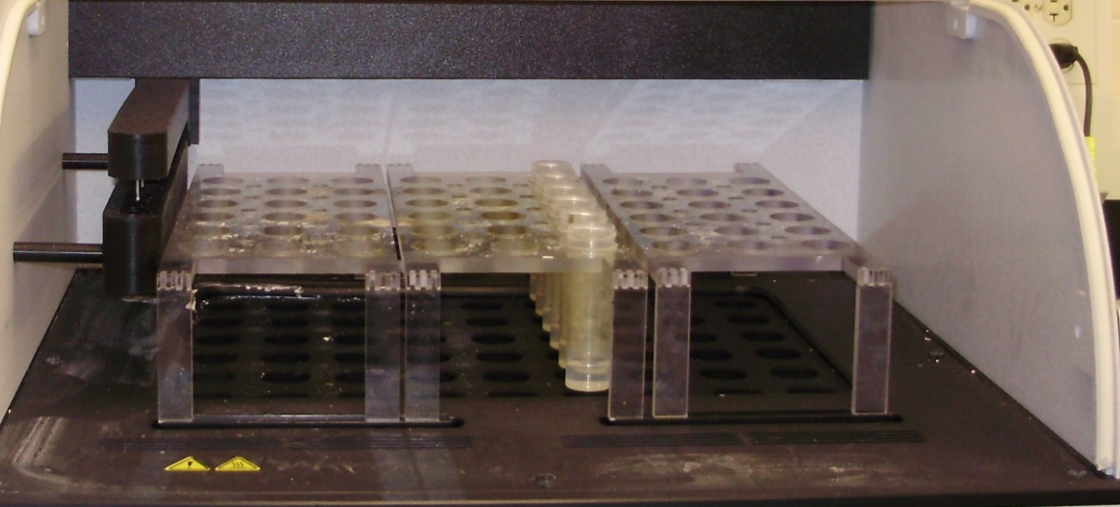
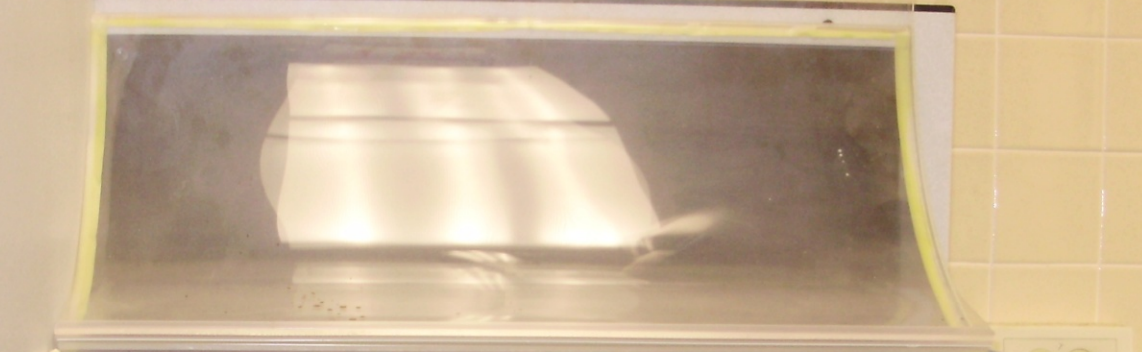
**Hot block**



- Mercury samples (industries, background, and sludge) are prepped automatically in the Environmental Express Autoblock II
- Analyzed by cold vapor analysis on a Leeman Labs Analyzer

Issue To / Émis à / A nombre de: \_\_\_\_\_  
Date / Date / Fecha: \_\_\_\_\_

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**ENVIRONMENTAL EXPRESS**



- Glassware for mercury, total metals and total phosphorous acid cleaned in the Milestone Traceclean





Verify acid level is above 50ml prior to use



### TRACECLEAN SAFETY TIPS

1. DO NOT OPERATE THIS UNIT WITHOUT A FULL CYCLE OF COMPLETION.
2. DO NOT OPERATE THIS UNIT WITH LESS THAN 500 ML OF ACID IN RESERVOIR.
3. NEVER ALLOWED REAGENTS OR SOLIDS ACCUMULATE IN VESSEL.
4. DO NOT OPERATE THIS UNIT UNLESS BATH HAS BEEN LOWERED COMPLETELY INTO FLASKS.
5. Wipe surface of lid with a damp paper towel, remove any items (CLEANED EQUIPMENT) FROM BATH.
6. NEVER ADD WATER TO OR REMOVE OPERATING UNIT.



traceCLEAN

Automatic cleaning system for Ultrasonic Chemistry™



- After all analysis is complete, samples are dumped, containers are cleaned and the process begins again.



➤ Questions?

