

Clean Techniques For Metals Collection And Analysis



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What Is Clean Technique?

- Purpose of clean technique is to prevent contamination
- “Clean” describes an item or environment where the background level of the target analyte is reduced (not necessarily eliminated) to a level that does not compromise the validity of the analysis
- Base of clean technique is proper sample collection

City's Metal History

- 1994 monitoring requirements:
 - 9 metals monitored
 - 7 metals parameters with discharge limits
- Metals violations:
 - 165 metals violations in 1994
 - 8 of these violations were for Hg

How Gastonia Implemented Clean Sampling and Analysis

- Identified potential sources of sample contamination
- Determined basic clean practices
- Established sampling equipment cleaning procedures
- Established sample collection procedures
- Established procedures for laboratory handling and analysis
- Identified equipment and supply needs for changes

Potential Areas of Contamination

- Sample collection equipment (dippers, jugs, bottles, samplers, strainers, etc...)
- Sampling location, environment
- Sample collection techniques
- Reagent quality (low grade acids)
- Laboratory techniques (digestions, handling, containers, etc...)

Basic Clean Practices

- Physically separate metals area
- Purchase new labware for metals use
- Keep all labware separate
- Avoid fans
- Don't sweep or dust during apparatus preparation or sample analysis
- Do not place objects into sample containers
- Do not wear gold jewelry during analysis

Equipment Cleaning Procedure

- Anything not known to be clean is assumed to be “dirty”
- Anything that comes into direct or indirect contact with the samples must be clean
- Wear clean room gloves and work in as clean an area as possible
- Use a dedicated non-metal brush
- Wash with hot water and a metal removing soap (e.g. Citranox)

Equipment Cleaning Procedure

- Rinse with DI water
- Rinse or soak with 1+1 nitric acid solution
- Rinse again with DI water
- Only place cleaned objects onto a new clean room wipe (e.g. TexWipe)
- Store cleaned objects in new plastic bags or clean sealed containers

Low Cost Methods for Sample Collection

- Education on sample contamination sources
- Clean/dirty hands technique for all metals sample collection
- Sampler strainers made in-house with PVC (no metal parts)
- Non-talc clean gloves for sample collection
- Collection of field reagent blanks

Clean Sampling Techniques

- Sampling site should be approached from downstream or downwind; sample should be taken facing upstream or upwind
- Avoid sampling near a bridge, in heavy wind or rain
- Samples should be collected by two people
 - Clean hands sampler (CH)
 - Dirty hands sampler (DH)

Clean Hands Sampler

- Responsibilities
 - Wear class 10 clean room gloves
 - All operations involving direct contact with sample or cleaned items
 - Removal of sample from sampler
 - Removal / replacing of container lids
 - Pouring up samples
 - Non-smoker

Dirty Hands Sampler

- Responsibilities
 - Wear class 10 clean room gloves
 - All operations not involving direct contact with the sample
 - Removing top of sampler
 - Operating peristaltic pump
 - Opening coolers

Composite Sampling Techniques

- Clean gloves are put on upon arrival at site
- DH removes lid and stops sampler
- CH removes sample container lid from plastic bag and places it on the carboy
- CH shakes carboy and pours sample into collection bottles
- CH places samples into the ice chest for transport to lab

Low Cost Methods for Clean Analysis Used by the Laboratory

- Education on sample contamination sources
- Wear non-talc clean gloves
- Cover metal surfaces
- Cleaning procedures for laboratory/equipment
- Air filters placed on HVAC vents
- Avoid use of paper towels (use clean room wipes)
- Store supplies in plastic bags, wrap or boxes

**Bagged Metals
Digestion
Equipment**



**Tile Board
Ceiling in
Metals Analysis
Room**

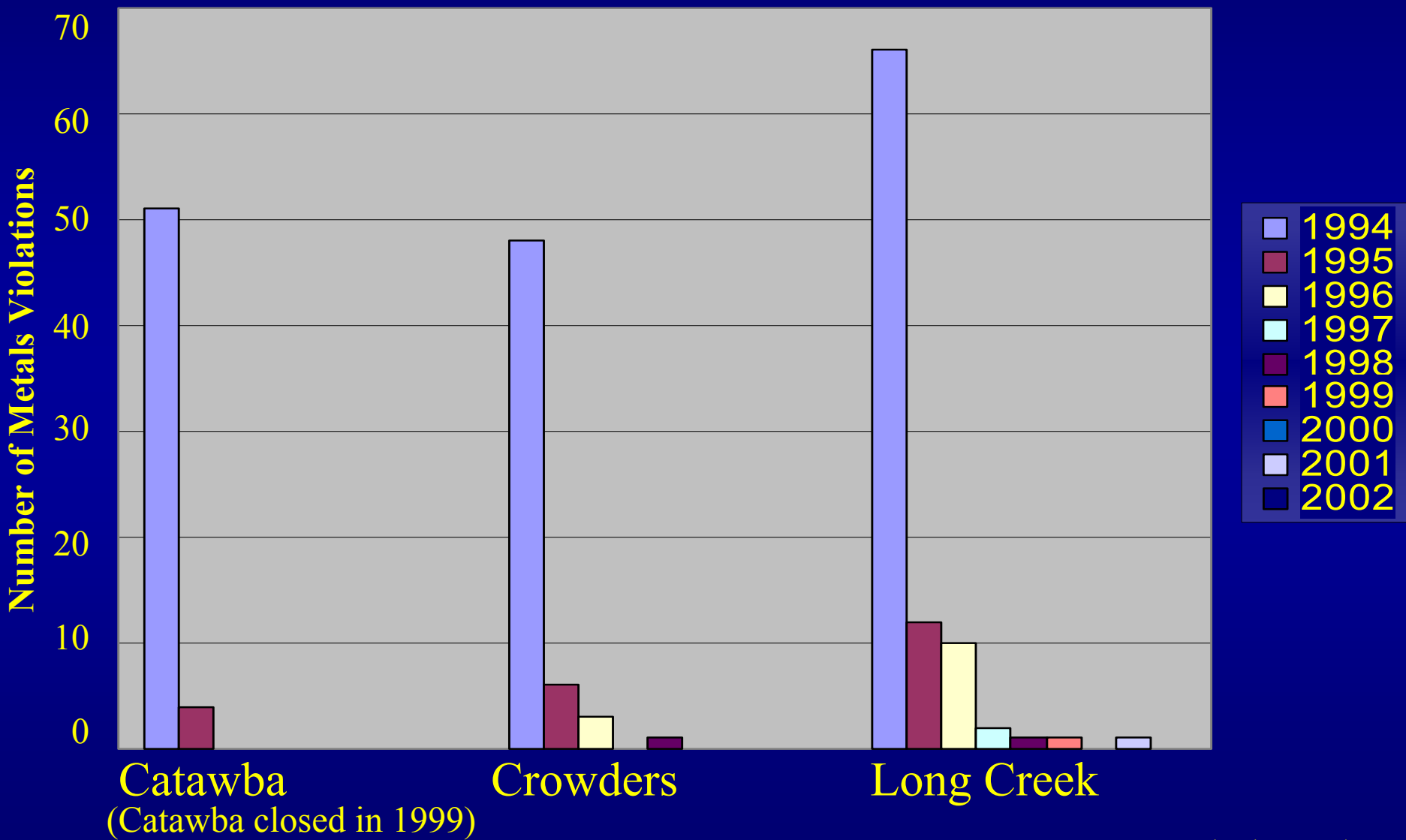
More Expensive Laboratory Modifications

- Replaced dropped ceiling panels with tile board
- Ultra high grade acids used in digestions
- No metal comes into contact with sample or reagents going into sample
- Duplicate all samples
- Clean bench/hood used for sample preparation

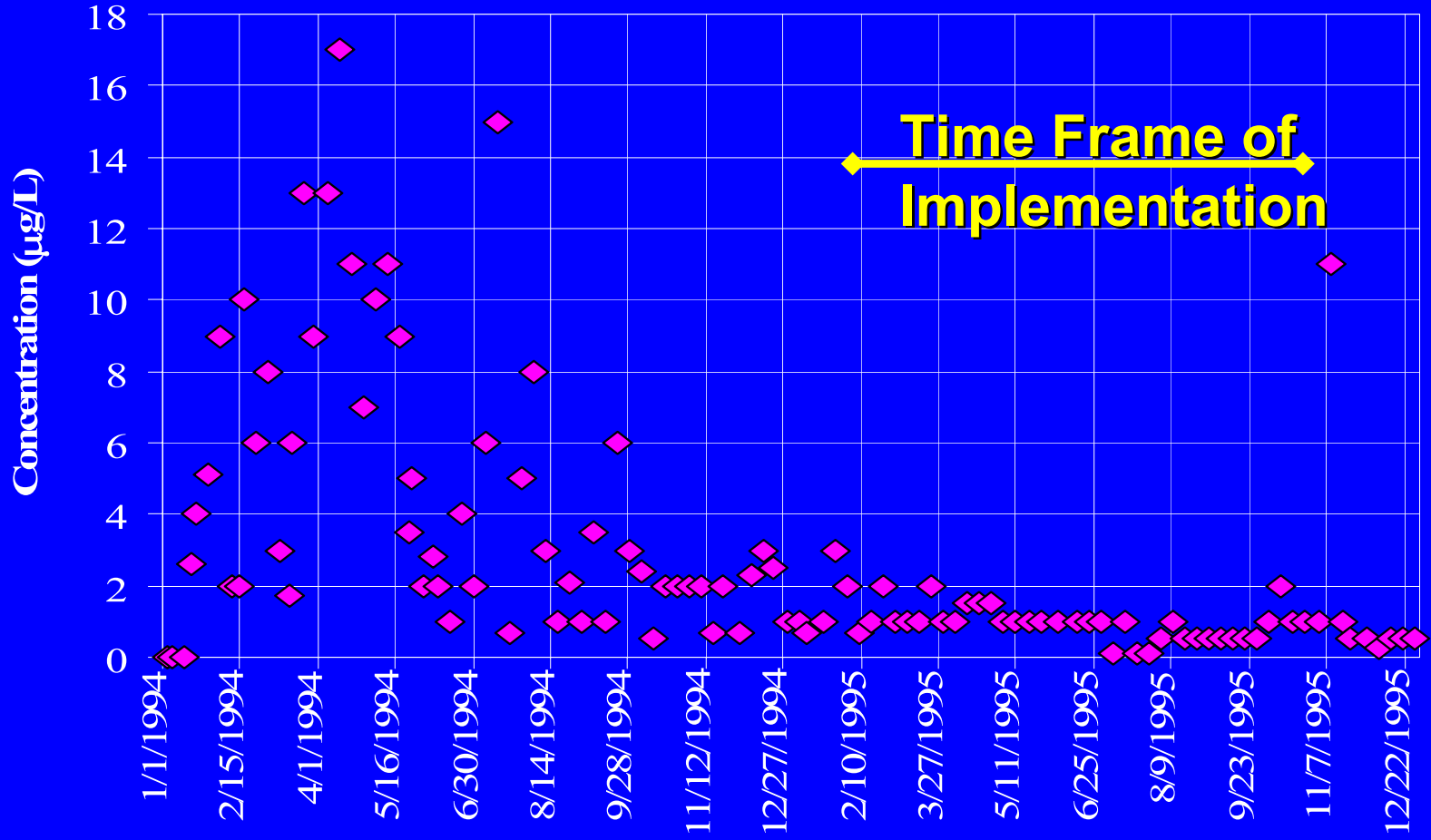


Sample Preparation in the Clean Hood

Reduction in Metals Violations at City of Gastonia Treatment Plants

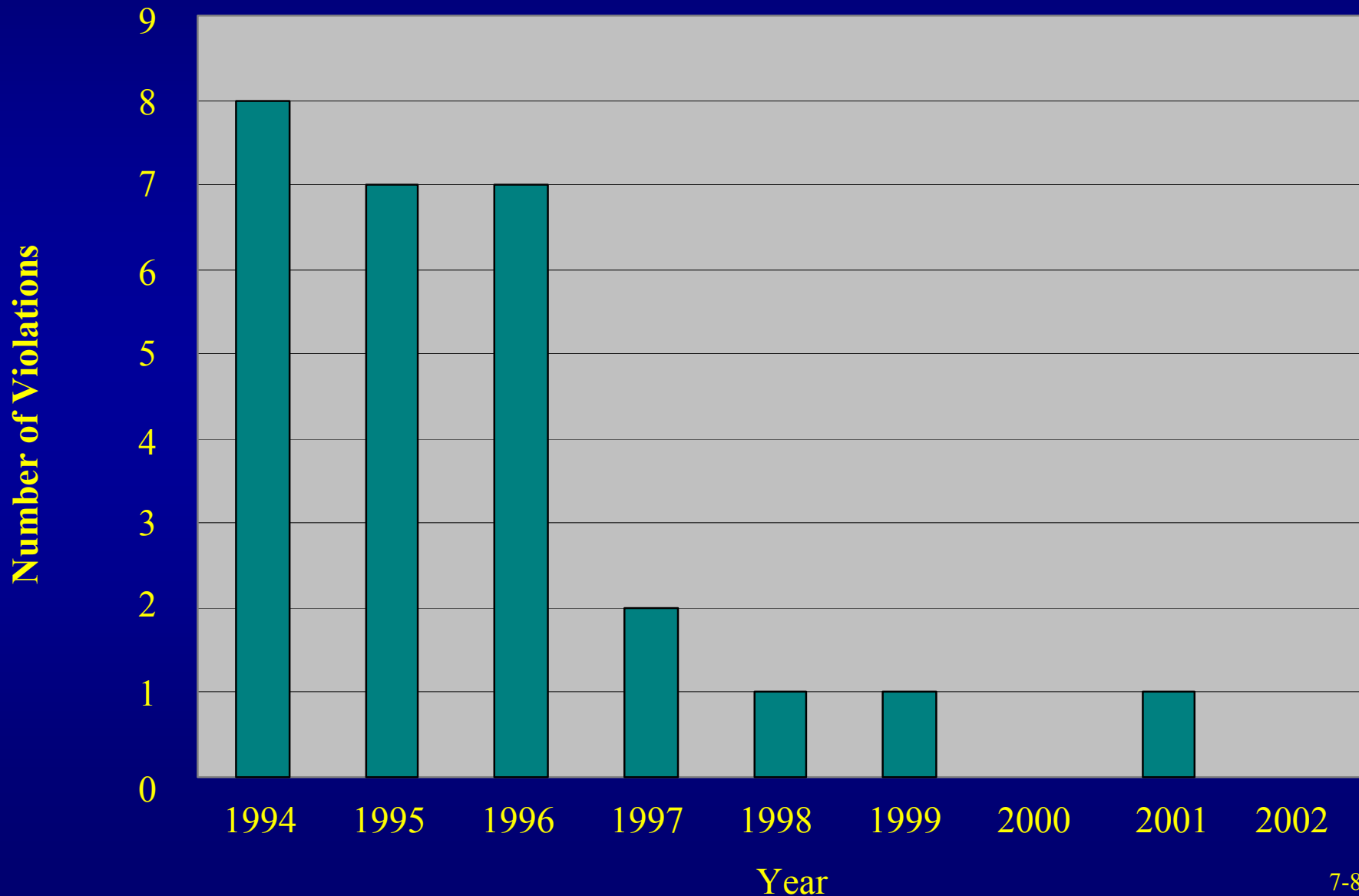


Cadmium



Metals decline at one of the WWTPs

Long Creek WWTP Mercury Permit Violations



Long Creek WWTP Mercury Permit Violations

Dec. 1997

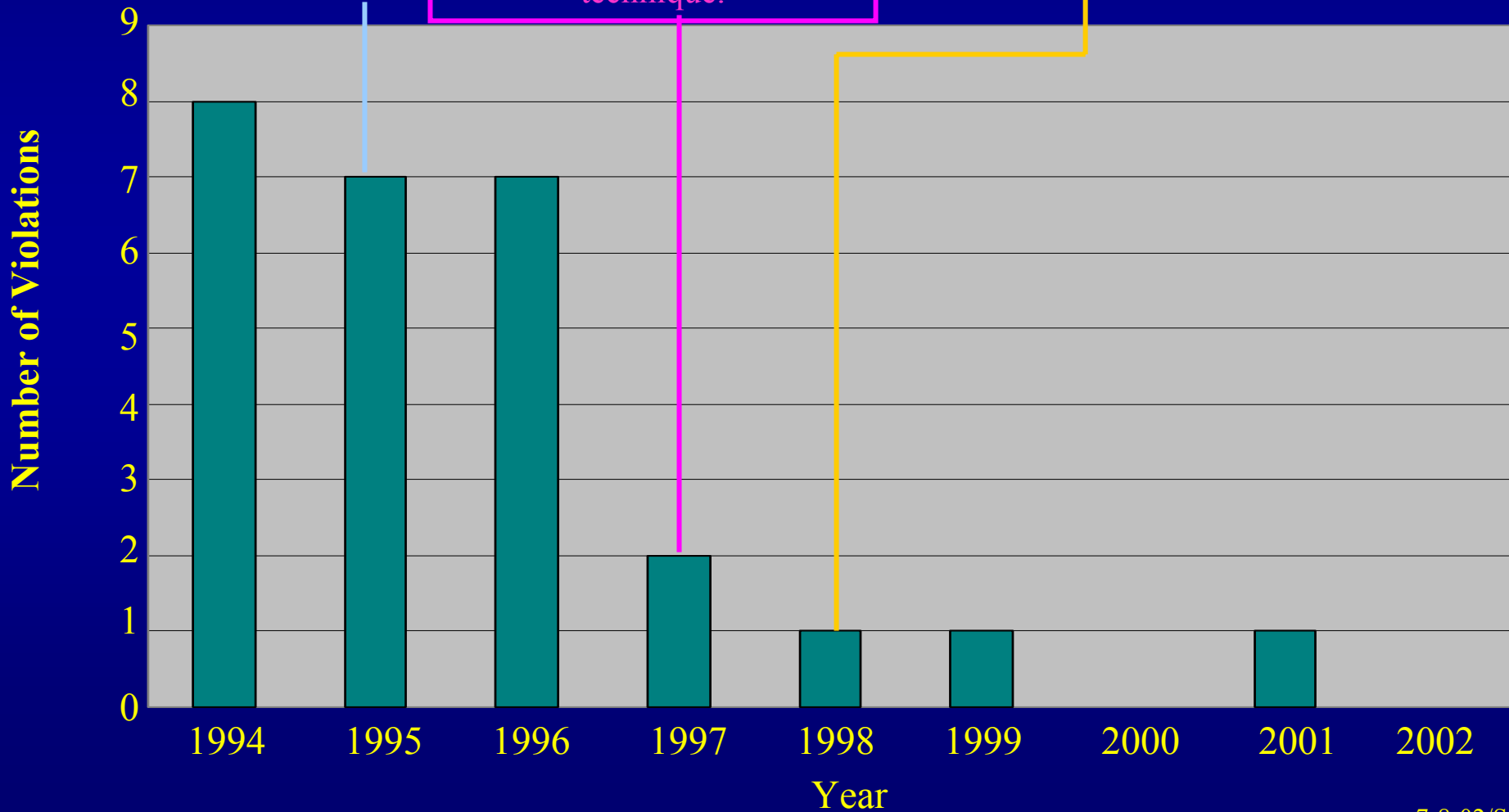
Switched from contract lab analysis without clean technique to in house analysis with clean technique.

June 1998

Switched from composite to grab sampling.

Feb. to Nov. 1995

Implemented clean sampling techniques.



What Has Gastonia Gained From Clean Sampling and Analysis ?

- We have essentially eliminated mercury violations
- We have achieved a more accurate representation of mercury concentrations in samples
- Data is more legally defensible