

# **Total Nitrogen Analysis Present and Future**

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NEXT WEEK

TOMORROW

OTHER DAY

**NOW**

SOMEDAY

IN THE FUTURE

NEXT YEAR

LATER

**Measure  
TKN**



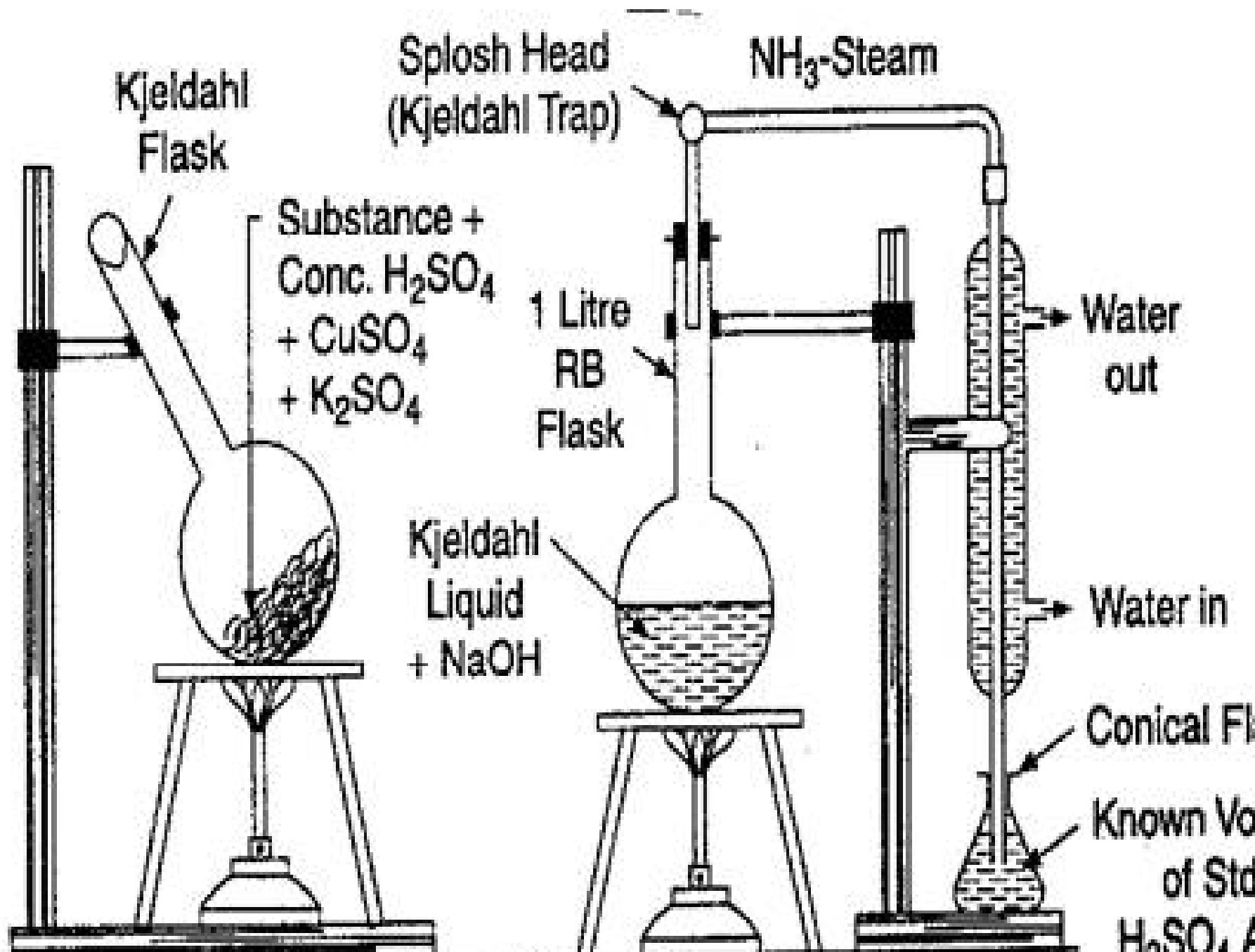
**Measure  
NO<sub>3</sub>/NO<sub>2</sub>-  
N**



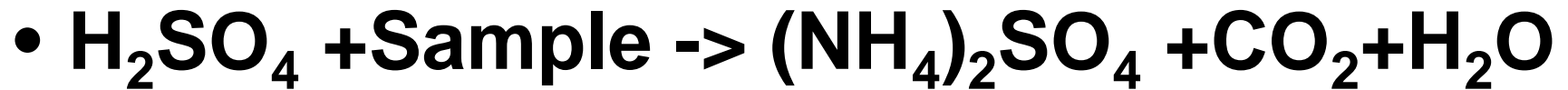
**TN**

H<sub>3</sub>

H<sub>3</sub>



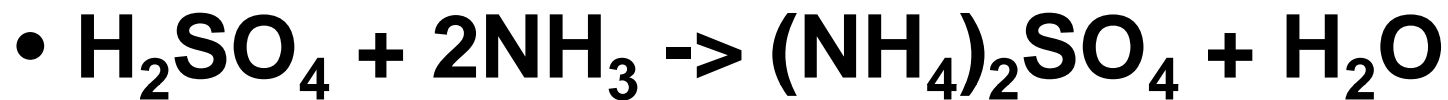
## Digestion



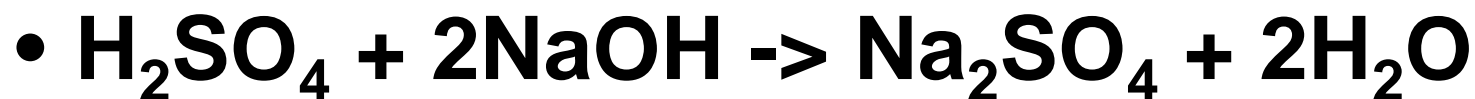
## Distillation



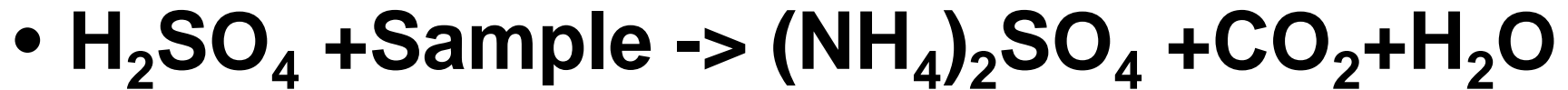
## Capture



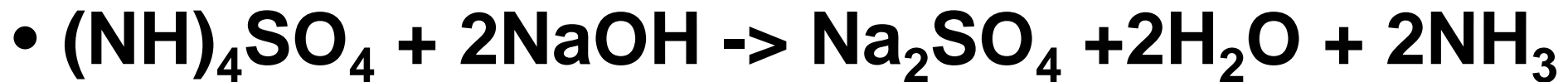
## Titration



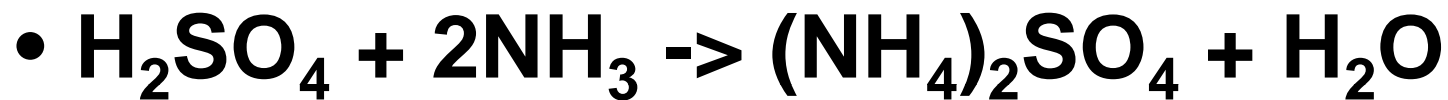
## Manual Digestion



## Distillation



## Capture



## Colorimetry

- Indophenol blue reaction with phenol

## TKN Methods

- Manual digestion
- Manual distillation
- Nessler, IC, Titration, ISE, phen

## TKN Methods

- Manual digestion
- Automated distillation/diffusion
- Direct Colorimetry

## NO<sub>3</sub>/NO<sub>2</sub> – N Methods

- IC, Reduction colorimetry

	Influent	Effluent
TKN (mg/L)	30	2
NO <sub>3</sub> -N (mg/L)	0	17
TN (mg/L)	30	19





$H_3$

12 FL OZ



**Sample Inject**



**NO out to  
detector**

**720 C reactor**

**TOC & TNb**

30

25

20

15

10

5

0

TKN TNb

Simple Nutrient

Complex Nutrient

Complex Nutrient

Simple N

H<sub>3</sub>



# Cons

# Pros

Requires special equipment

Particulates?

Complete Oxidation

Detects all N compounds

Detects TOC

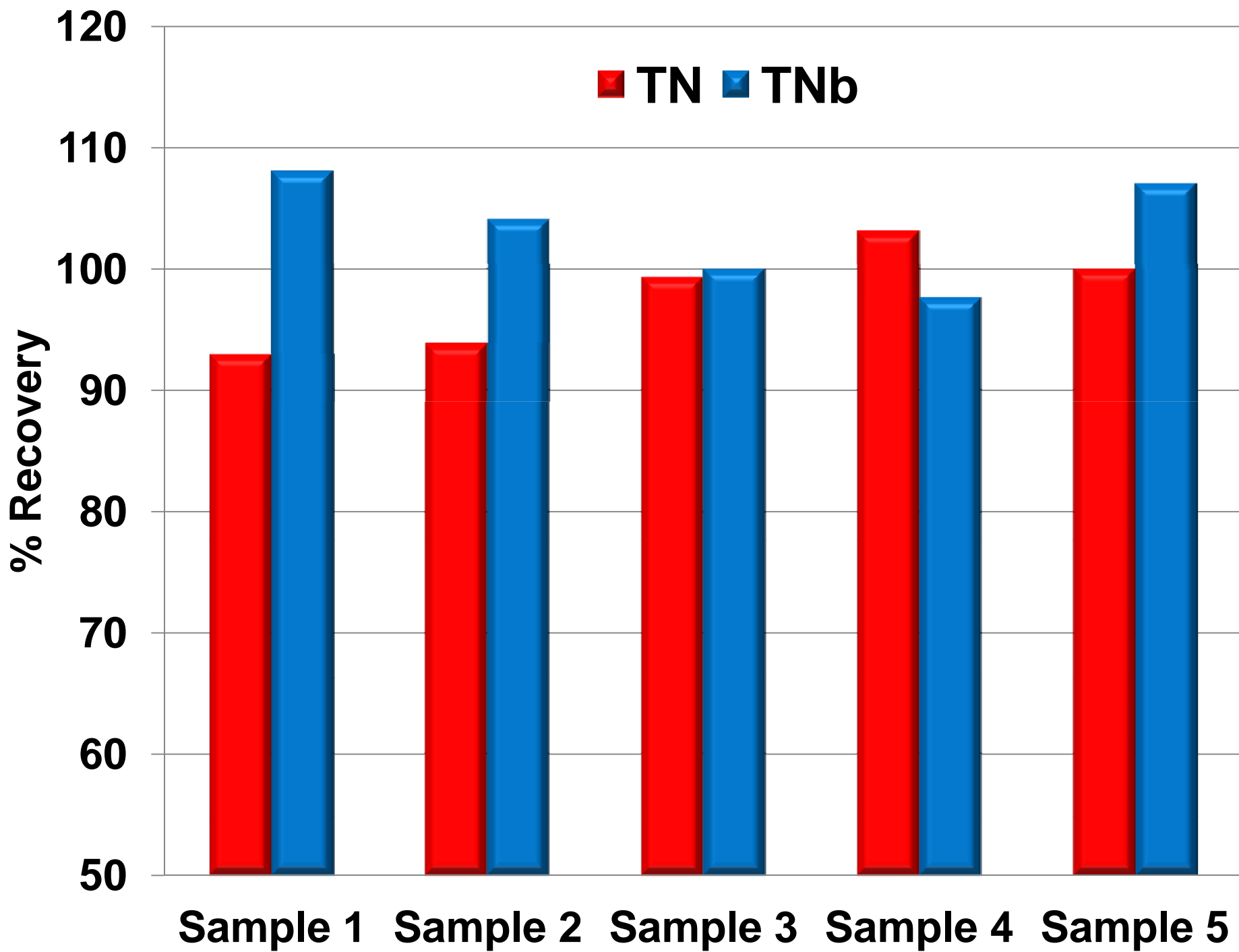
H<sub>3</sub>



**Manually digests**

**Determine  $\text{NO}_3^-$**

**Measures all T**



	<b>TOC (mg/L)</b>	<b>TKN + NOx (mg/L)</b>	<b>TN (mg/L)</b>
<b>Cecil</b>	<b>17.5</b>	<b>2.9</b>	<b>2.9</b>
<b>Greenville 1</b>	<b>36.0</b>	<b>3.1</b>	<b>3.5</b>
<b>Hiwassee 2</b>	<b>59.0</b>	<b>8.8</b>	<b>8.9</b>
<b>Irwin</b>	<b>108</b>	<b>17.7</b>	<b>16.9</b>



	<b>TKN (mg/L)</b>	<b>TKN (%RSD)</b>	<b>TN (mg/L)</b>	<b>TN (%RSD)</b>
<b>Bear Creek</b>	<b>0.18</b>	<b>10.65</b>	<b>0.22</b>	<b>5.7</b>
<b>Silver Fork</b>	<b>0.36</b>	<b>19.29</b>	<b>0.41</b>	<b>6.4</b>
<b>Salt River</b>	<b>0.59</b>	<b>25.31</b>	<b>0.76</b>	<b>3.2</b>
<b>Ted Shanks</b>	<b>0.61</b>	<b>25.25</b>	<b>1.05</b>	<b>6.0</b>

# persulfate

- **Manual digestion**
- **Colorimetric Detection**
  - **Dimethylphenol (or similar)**
  - **Direct UV**
  - **Reduce  $\text{NO}_3$  to  $\text{NO}_2$**
  - **Ion Chromatography**

# TN persulfate methods

**Cons**

**Pros**

Requires  
manual  
digestion

TOC?

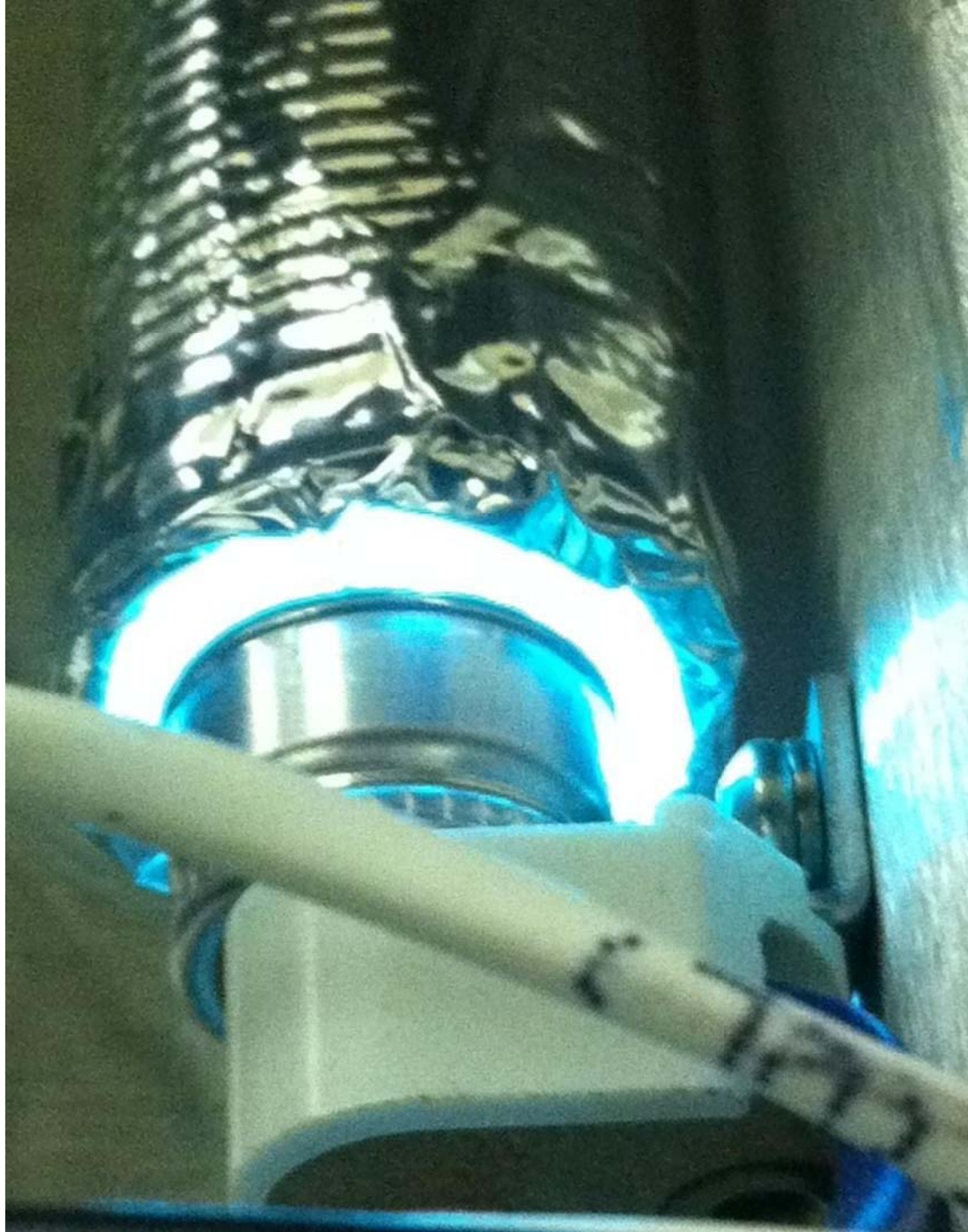
Complete  
Oxidation

Detects all N  
compounds

Simultaneous  
TP

H<sub>3</sub>

# Digestions

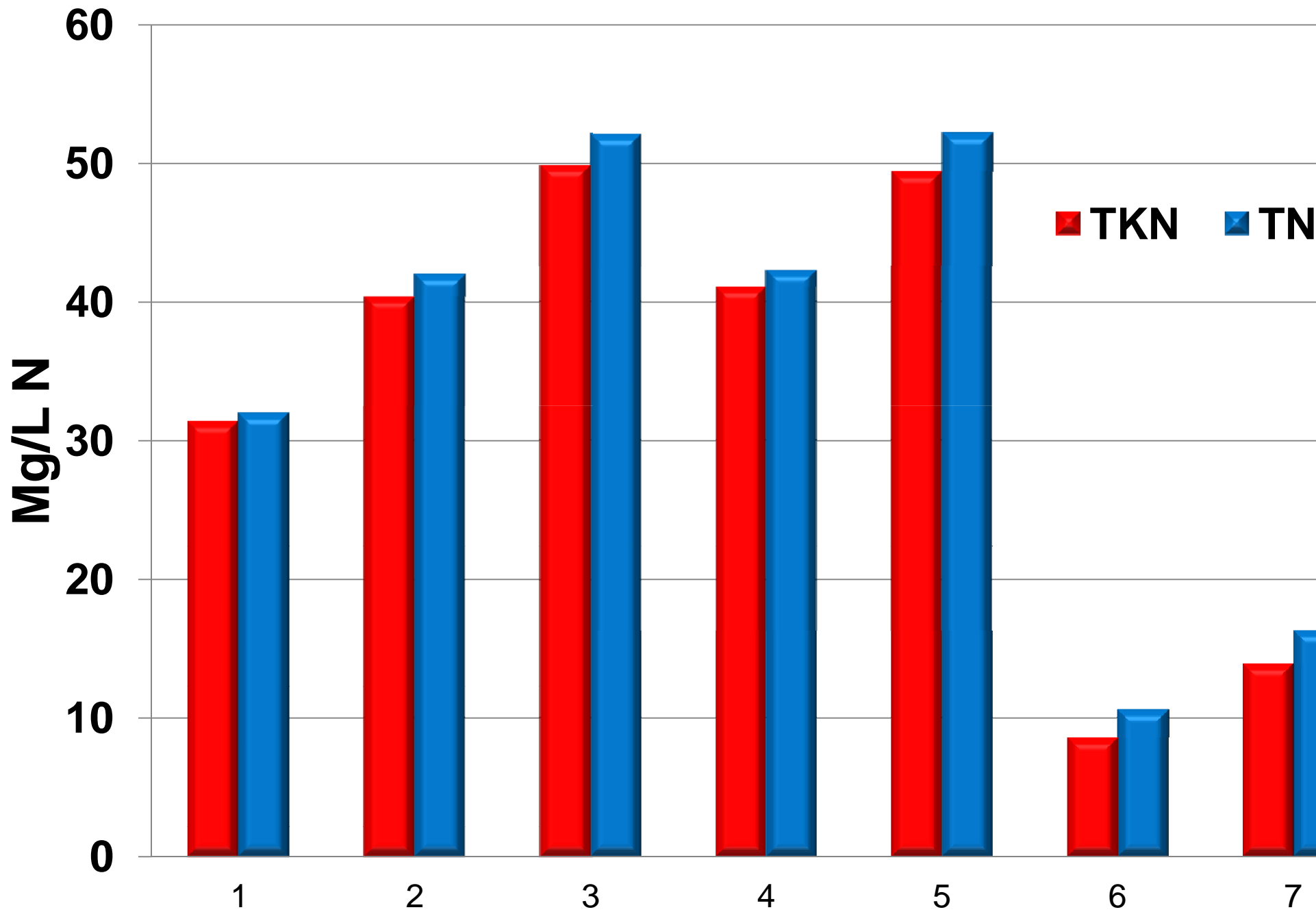


**Automated digestion**

**Determine  $\text{NO}_3^-$**

**Measures all T**

$\text{H}_3$



# Techniques

- **Segmented Flow or Flow Injection**
- **Colorimetric Detection**
  - **Direct UV**
  - **Reduce  $\text{NO}_3$  to  $\text{NO}_2$**

# Automated UV Alkaline Digestion



**Particulates?**

# automated TN persulfate methods

**Cons**

**Pros**

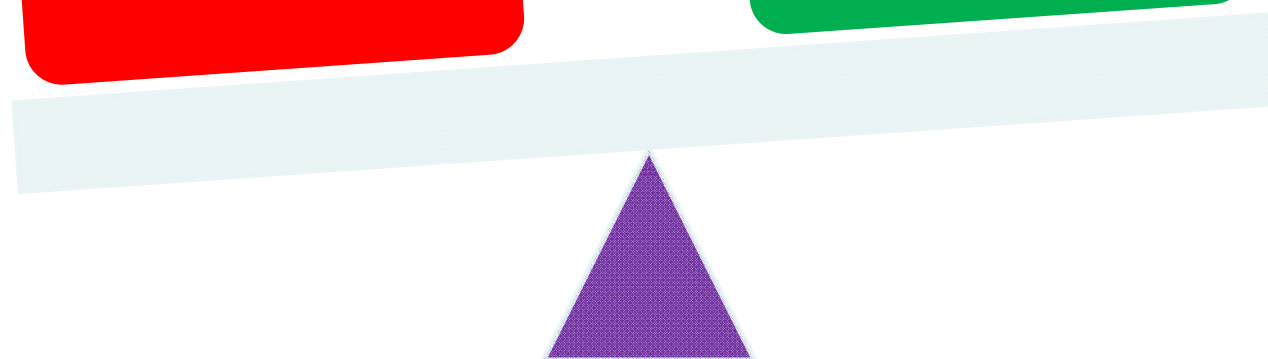
**Simultaneous  
TP?**

**Solids?**

**TOC?**

**Complete  
Oxidation**

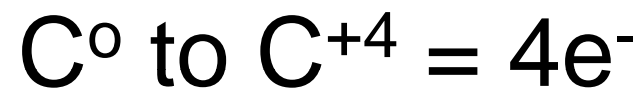
**Detects all N  
compounds**





# methods

- **Organic carbon loading?**
- **Particulate solids?**
- **No EPA parameter**
- **No EPA approved method**
- **Persulfate contains Nitrogen**



**About 100 ppm C upper limit**

# hard to sample

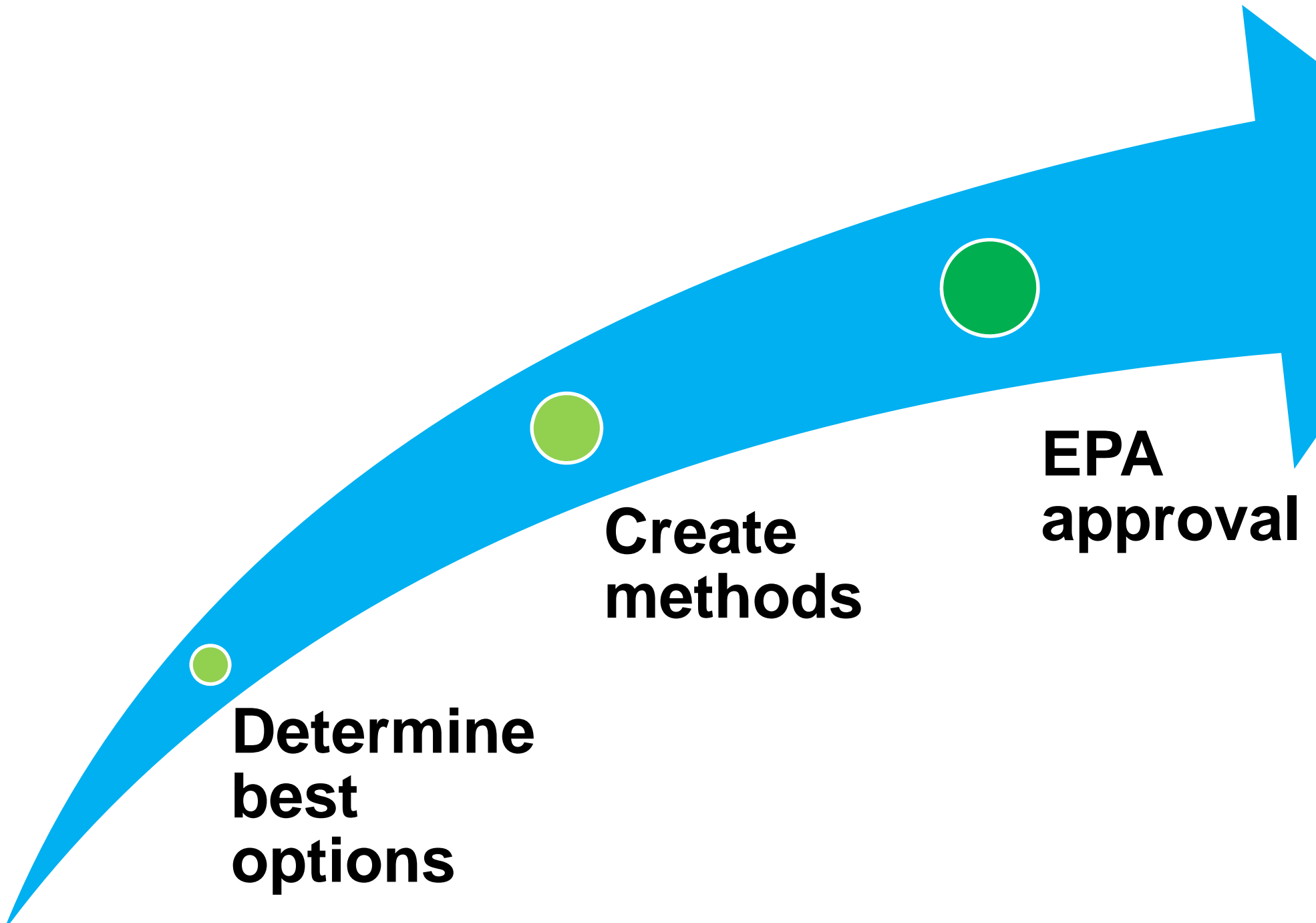


**~ 15 - 20% N attached to particulates**

**Particulates > ~ 30 ppm TSS N/A by CFA**

**Non quantitative transfer of particulates  
to HTCO**

H<sub>3</sub>



**Determine  
best  
options**

**Create  
methods**

**EPA  
approval**

H<sub>3</sub>

**William Lipps**  
**[www.oico.com](http://www.oico.com)**