

# Enhanced Productivity ICP (EP-ICP) for Tribology Applications

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Glass Expansion

# Importance of metal analysis

Element	Wear metal	Contaminant	Additive
Cu	<input checked="" type="checkbox"/>		
Fe	<input checked="" type="checkbox"/>		
Pb	<input checked="" type="checkbox"/>		
Cd	<input checked="" type="checkbox"/>		
Al	<input checked="" type="checkbox"/>		
Cr	<input checked="" type="checkbox"/>		
B		<input checked="" type="checkbox"/>	
Si		<input checked="" type="checkbox"/>	
Na		<input checked="" type="checkbox"/>	
K		<input checked="" type="checkbox"/>	
Ca			<input checked="" type="checkbox"/>
P			<input checked="" type="checkbox"/>
Zn			<input checked="" type="checkbox"/>

# Two flow injection approaches

## Niagara Plus

- Off-line dilution with Hamilton ML500 series
- Niagara Plus flow injection accessory
- 18.5 sec/sample

## Assist

- All in one dilution and analysis
- Assist flow injection accessory (syringe-based)
- AIM high capacity autosampler
- 28 sec/sample

# Niagara II valve characteristics

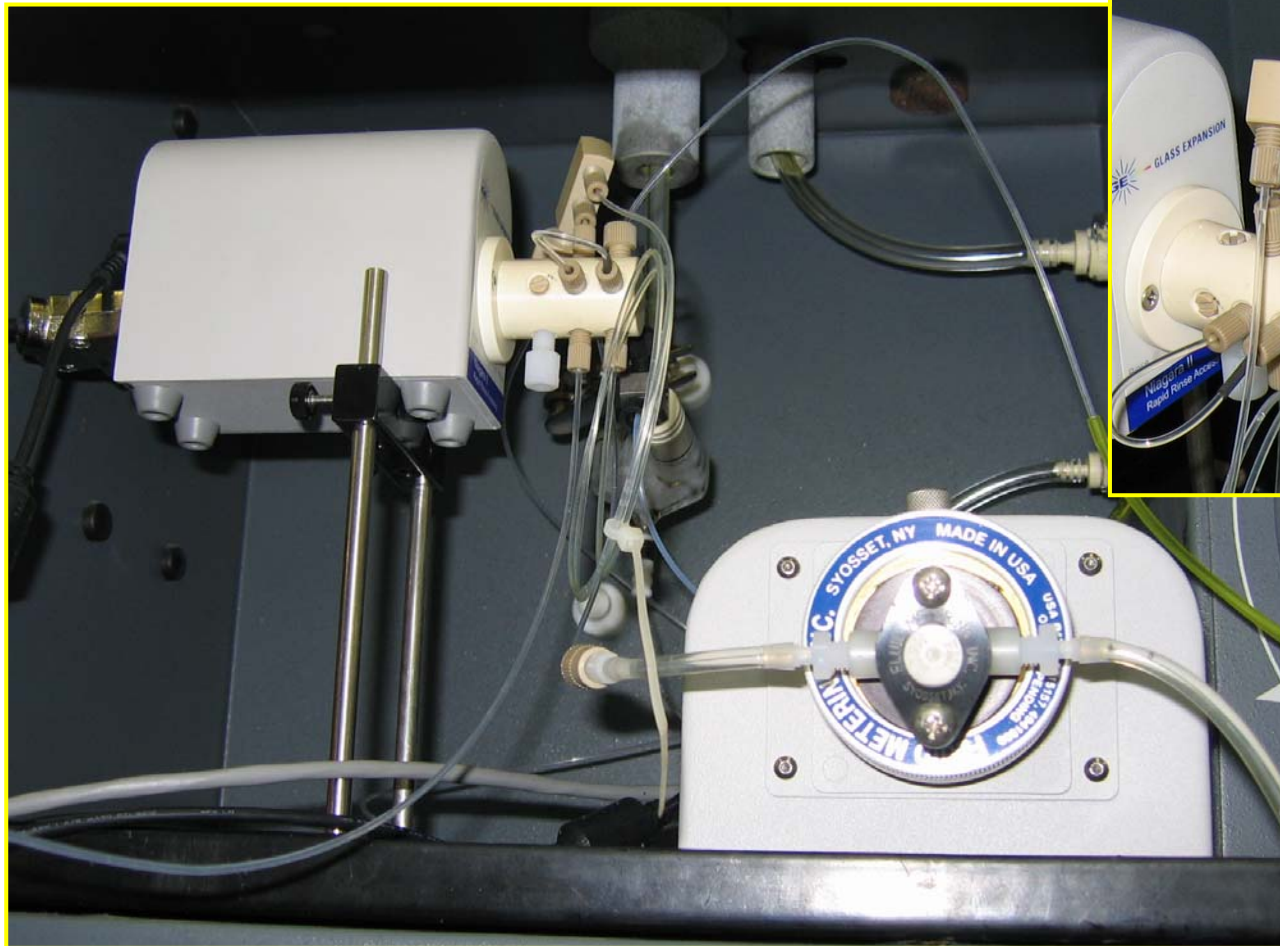
- Minimum swept volume
- Minimum distance from nebulizer
- Constant path ID
- Rigid rotor
- Removable stator
- Easy to configure



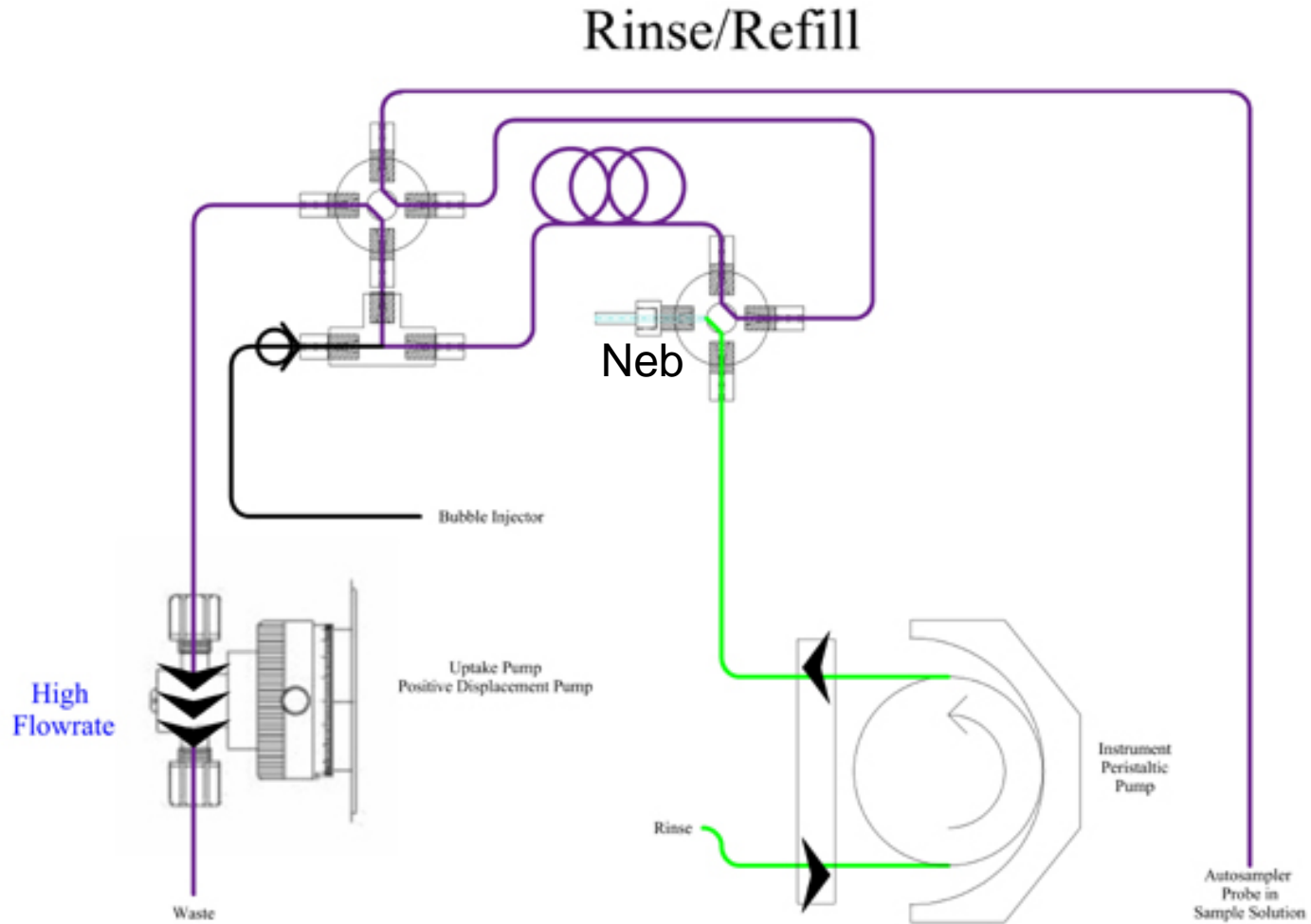
# Experimental parameters

- Varian Vista Pro radial ICP-OES
- Twinnabar spray chamber (20ml internal volume with baffle)
- Ceramic VeeSpray nebulizer
- Single-piece quartz torch with 0.8mm bore injector

# Niagara Plus on Varian Vista ICP

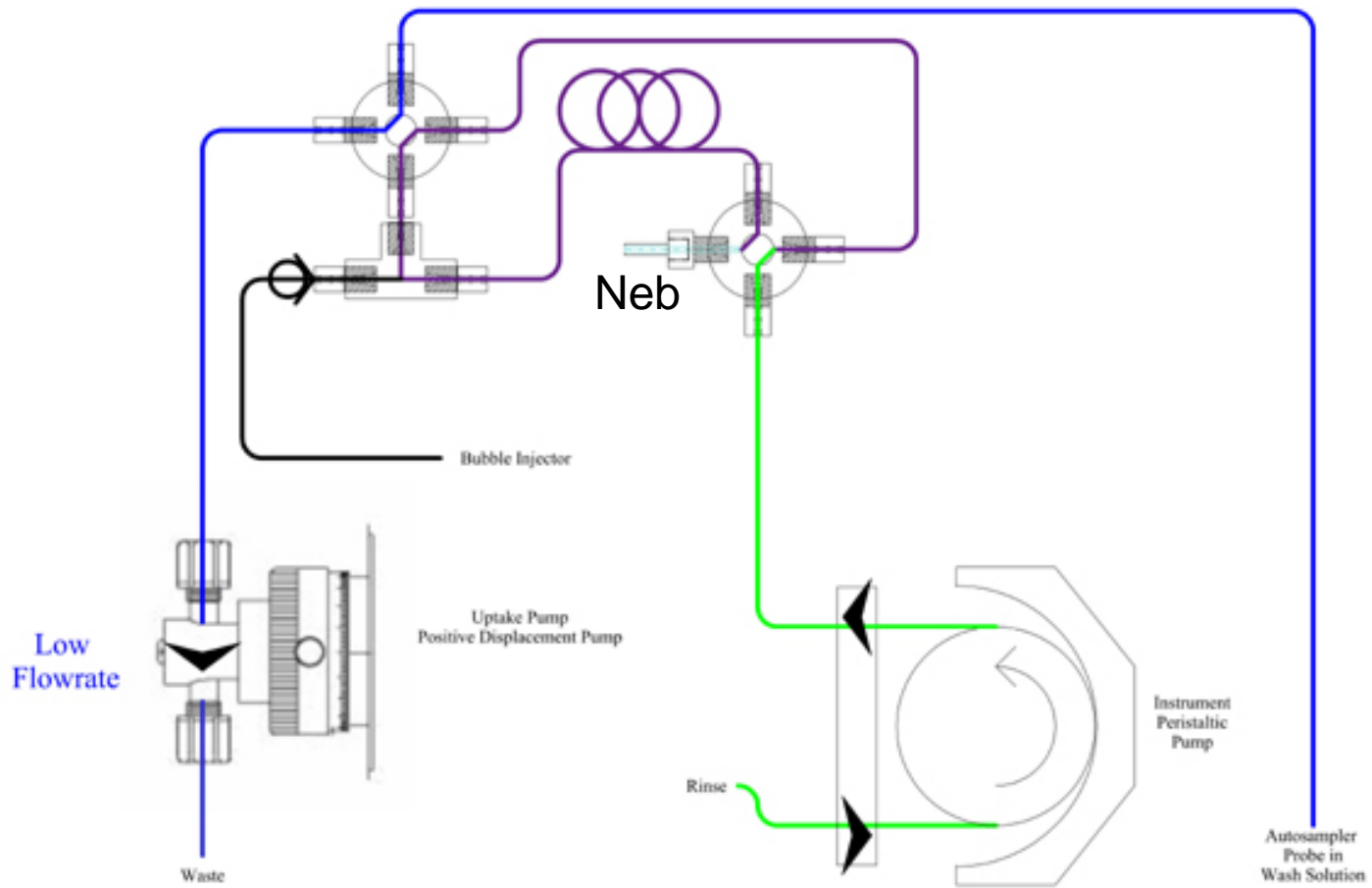


# Niagara Plus schematic



# Niagara Plus schematic

## Home/Inject





# Instrument method

## Niagara Plus method

## Niagara method

**Setup**

Method settings | Standby mode | Communications

Method: N2Plus4sec [v] [New method]

Loop uptake delay: 4.5 sec [Save method]

Uptake pump speed (Refill): 350 rpm [Save As]

Uptake pump speed (Inject): 220 rpm

Time in sample: 3.5 sec

Bubble inject time: 4.4 sec

Convert inches to mm: [ ] Inches = 0.00 mm [OK] [Cancel]

**Method Editor - Results Exist**

File Edit View Graphics Options Tools QC Tests Help!

Element | Conditions | Standards | Notes

Conditions set

Element	Retention Time	Color
Ag 328.068	328.068	Green
Al 308.215	308.215	Green
B 249.678	249.678	Green
Ba 493.408	493.408	White
Ca 317.933	317.933	Green
Cd 226.502	226.502	Yellow
Cr 267.716	267.716	Red
Cu 324.754	324.754	Green
Fe 238.204	238.204	Green
K 766.491	766.491	Green
Mg 285.213	285.213	White
Mn 257.610	257.610	White
Mo 202.032	202.032	Green
Na 588.995	588.995	Red
Ni 231.604	231.604	White
P 177.434	177.434	White
Pb 220.353	220.353	White
S 181.972	181.972	Green
Sb 217.582	217.582	White
Si 251.611	251.611	White
Sn 283.998	283.998	White
Ti 336.122	336.122	White
V 292.401	292.401	Green

Conditions used by: All Lines

Power (kW): 1.40

Plasma flow (L/min): 18.0

Auxiliary flow (L/min): 2.25

Nebulizer flow (L/min): 0.75

Replicate read time (s): 2.00

Instr stabilization delay (s): 4

Sample introduction settings

Sample uptake delay (s): 0

Pump rate (rpm): 15

Rinse time (s): 0

Fast pump (Samp delay/rinse)

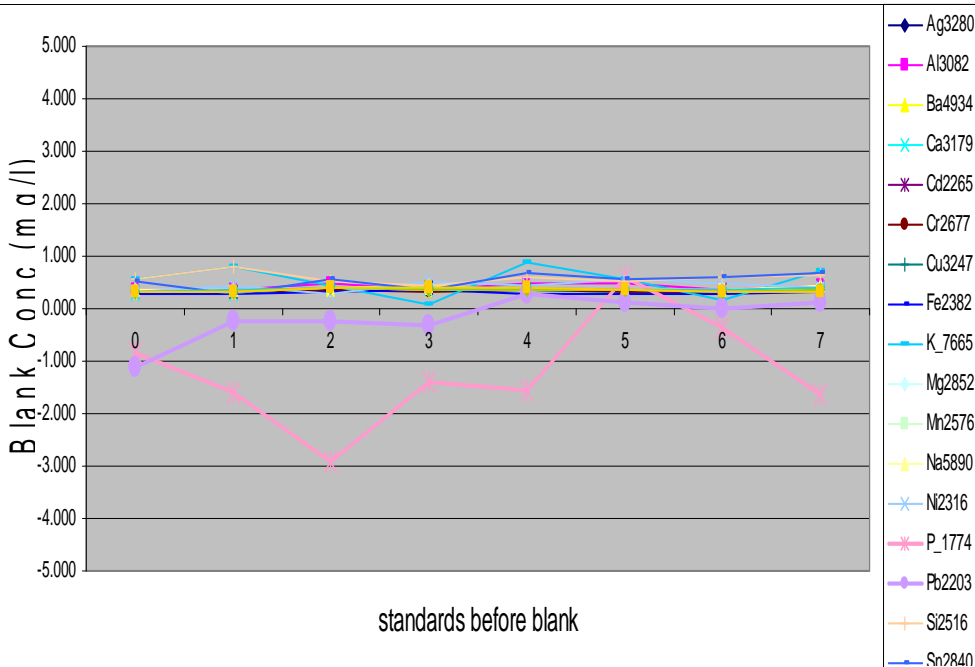
Smart Rinse [Smart Rinse...]

General settings

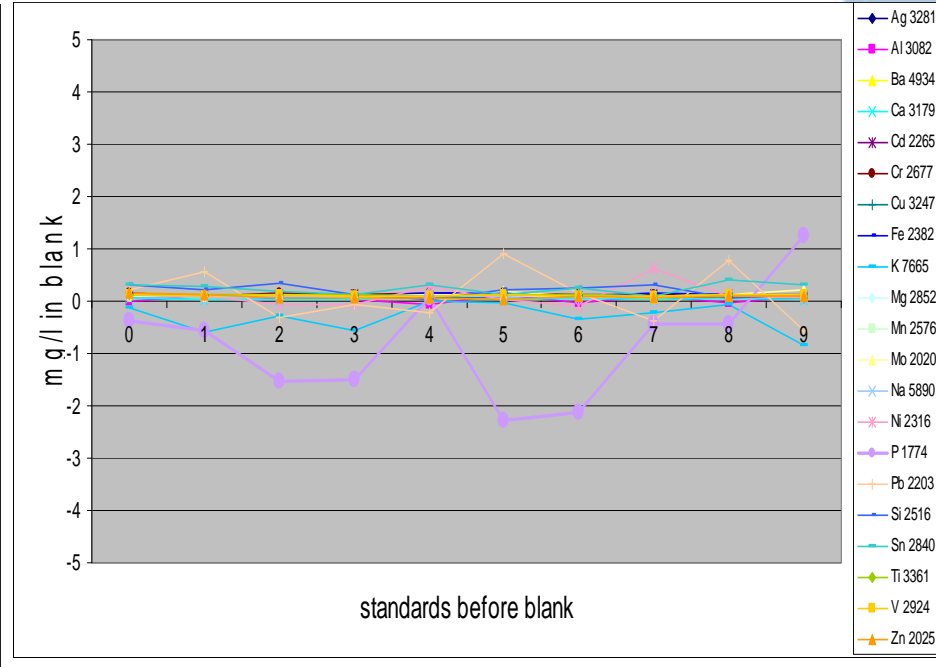
Replicates: 2

# Carryover Comparison (after 100mg/L standards)

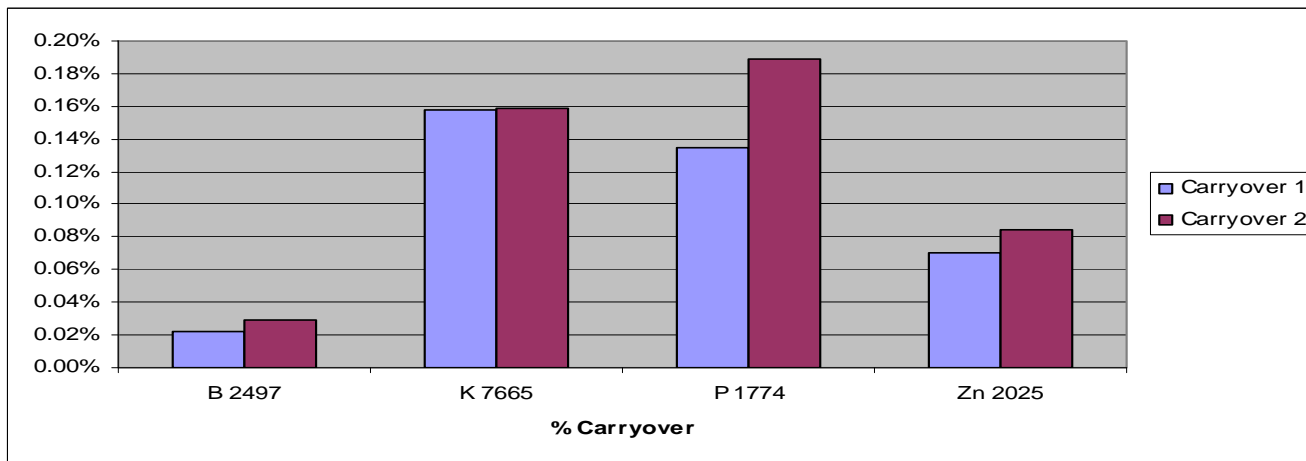
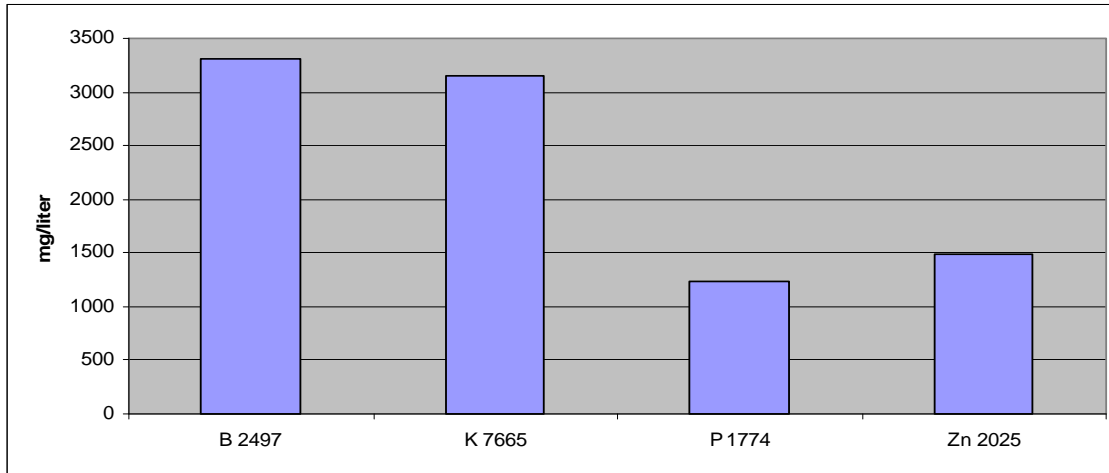
## Diluted oil without rinse



## Diluted oil with 1s rinse



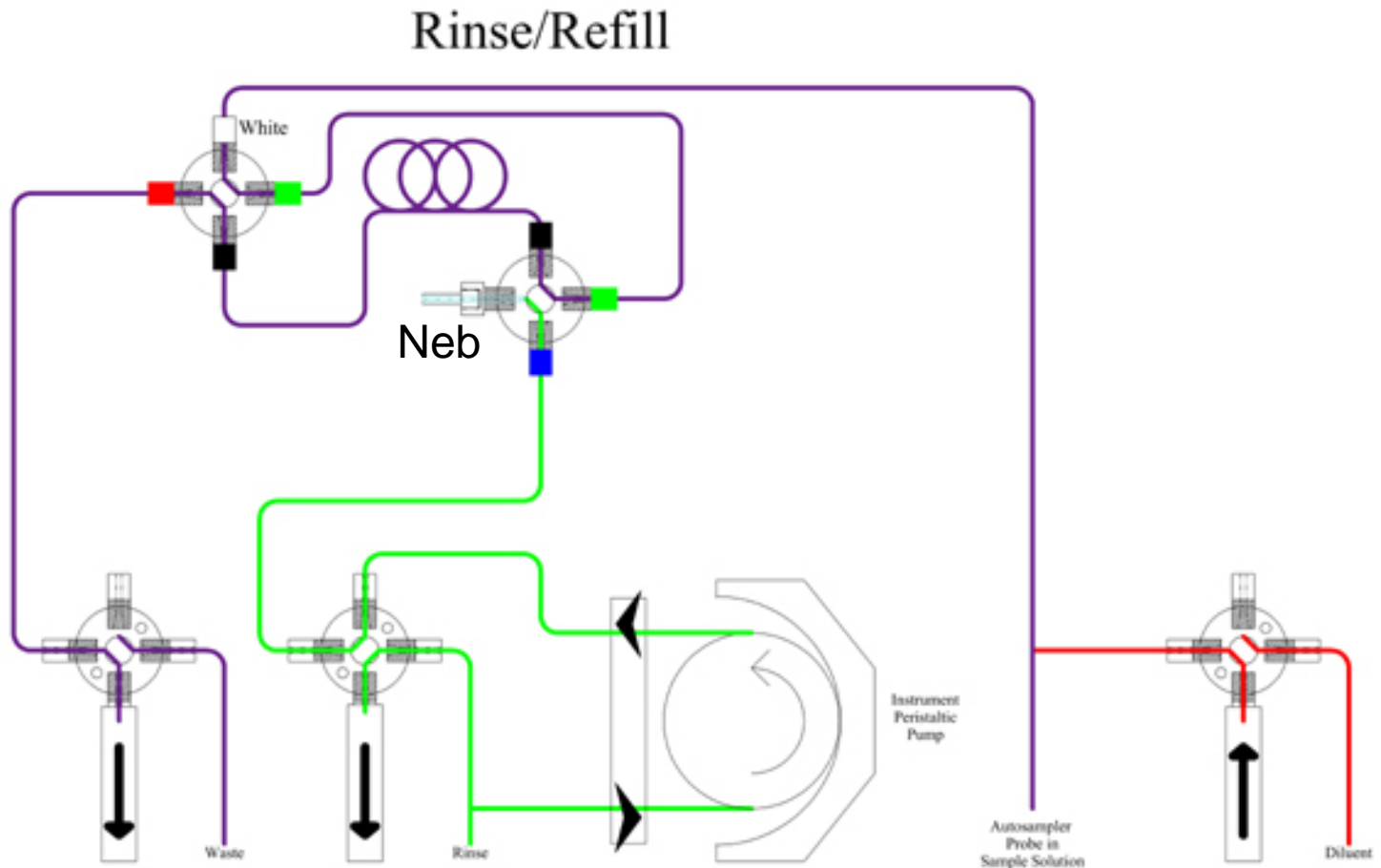
# Carryover in diluted oil samples 1s rinse



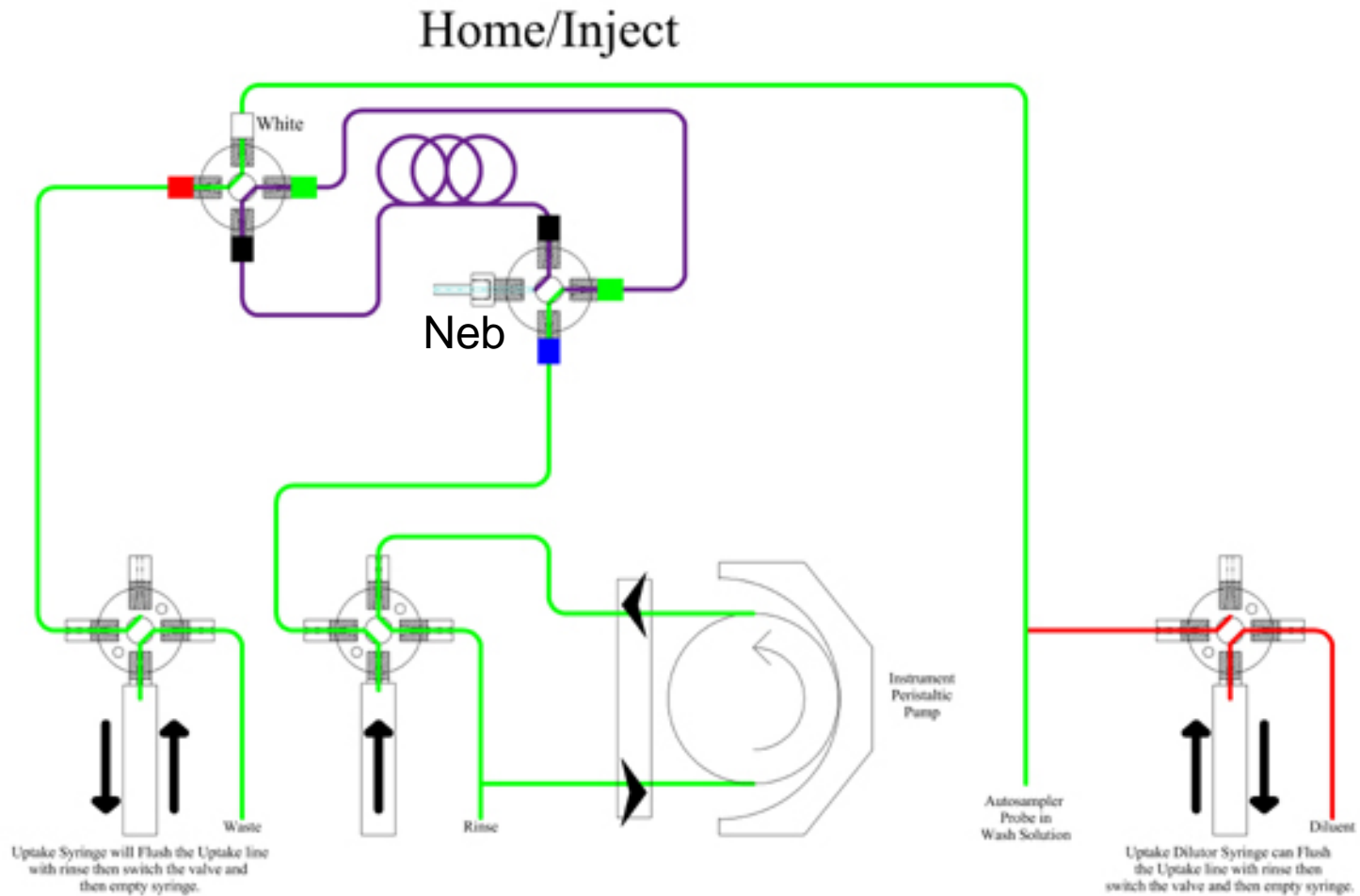
# Assist on Varian Vista Pro



# Assist Schematic- rinse/refill



# Assist Schematic- home/inject



# Assist method

**Setup**

Method settings | Sample syringe | Uptake syringe | Standby mode | Communications

Method: ALSolvsVirginia

**Assist settings**

Loop uptake delay: 15 sec  
Purge time: 0 sec  
Purge rate: 0 mL/min  
Syringe refill time: 5 sec  
Uptake pump speed (Refill): 100 rpm  
Uptake pump speed (Inject): 50 rpm  
Uptake pump speed (Move): 100 rpm  
Time in sample: 11 sec  
Bubble inject time: 14 sec

**Sample introduction settings**

Nebulizer flow rate: 1.75 mL/min  
Total read time: 4 sec  
Stabilization/Read delay: 4 sec  
% of diluent: 0 %

Total volume injected: 0.233 mL

Convert inches to mm:  Inches = 0.00 mm

**Setup**

Method settings | Sample syringe | Uptake syringe | Standby mode | Communications

**Configuration**

Barrel volume: 2.50 mL  
A barrel size of 0.5mL to 25ml should be used.  
Possible flow rates using this barrel size  
Min possible flow rate is 0.125 mL/min  
Max possible flow rate is 150 mL/min

**Priming**

No. of priming cycles: 3  
Priming flow rate: 2 mL/min

Refill rate: 29.688 mL/min  
Injection rate: 1.75 mL/min  
Total volume used: 0.233 mL

mm

**Setup**

Method settings | Sample syringe | Uptake syringe | Standby mode | Communications

**Sample Uptake**

Barrel volume: 10.00 mL  
A barrel size of 10mL to 25ml should be used.  
Sample uptake volume: 3.5 mL  
Possible flow rates using this barrel size  
Min possible flow rate is 0.5 mL/min  
Max possible flow rate is 600 mL/min  
Sample uptake rate: 15 mL/min  
Sample empty rate: 580 mL/min  
Minimum loop uptake delay is 14 sec  
% Uptake dilution: 90 %  
Dilutor barrel volume: 10.00 mL  
A barrel size of 5mL to 25ml should be used.  
Dilutor refill rate: 50 mL/min

**Uptake Line Flush**

Enable line flush  
Flush volume: 2 mL  
Flush rate: 35 mL/min  
 Enable dilutor during flush

**Loop rinse**

Enable rinse  
Rinse volume: 0 mL  
Rinse rate: 0 mL/min  
Time required: NaN sec

Convert inches to mm:  Inches = 0.00 mm

**Method Editor - Results Exist**

File Edit View Graphics Options Tools QC Tests Help!

Element Conditions Standards Notes

Conditions set

Ag	328.068	328.068	Green
Al	308.215	308.215	Grey
Ba	493.408	493.408	White
Ca	317.933	317.933	Green
Cd	226.502	226.502	Yellow
Cr	267.716	267.716	Red
Cu	324.754	324.754	White
Fe	238.204	238.204	Brown
K	766.491	766.491	Green
Mg	285.213	285.213	White
Mn	257.610	257.610	Grey
Mo	202.032	202.032	Green
Na	588.995	588.995	Red
Ni	231.604	231.604	Grey
P	177.434	177.434	White
Pb	220.353	220.353	Grey
Si	251.611	251.611	Red
Sn	283.998	283.998	Grey
Ti	336.122	336.122	Grey
V	292.401	292.401	Brown
Zn	202.548	202.548	Green

Conditions used by: All Lines

Power (kW): 1.40

Plasma flow (L/min): 18.0

Auxiliary flow (L/min): 2.25

Nebulizer flow (L/min): 0.75

Replicate read time (s): 2.00

Instr stabilization delay (s): 13

Sample introduction settings

Sample uptake delay (s): 0

Pump rate (rpm): 30

Rinse time (s): 0

Fast pump (Samp delay/rinse)

Smart Rinse Smart Rinse...

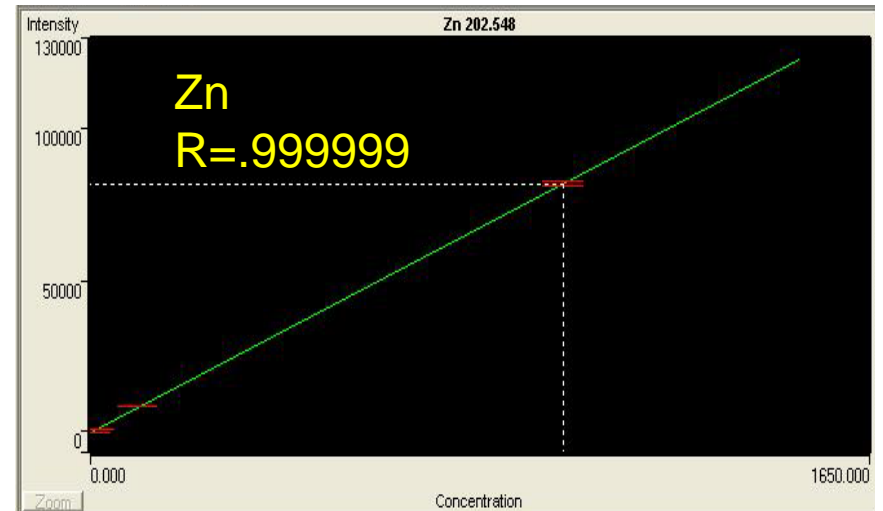
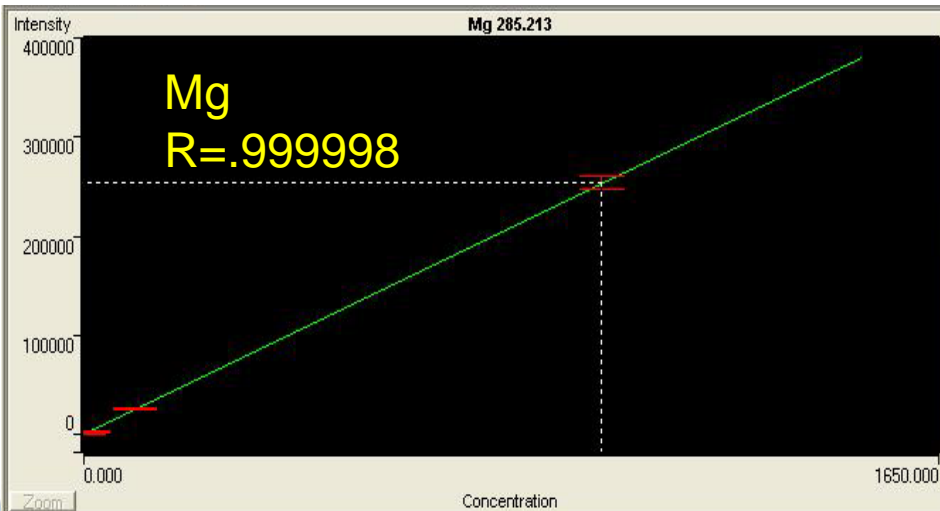
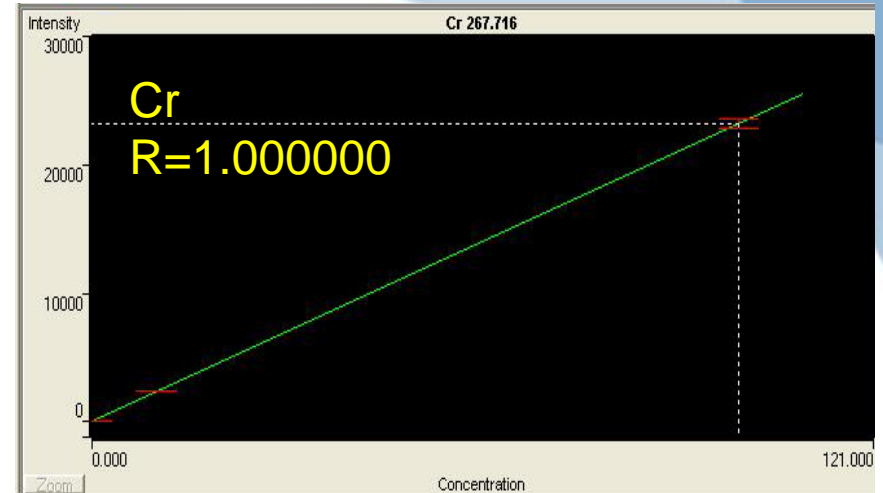
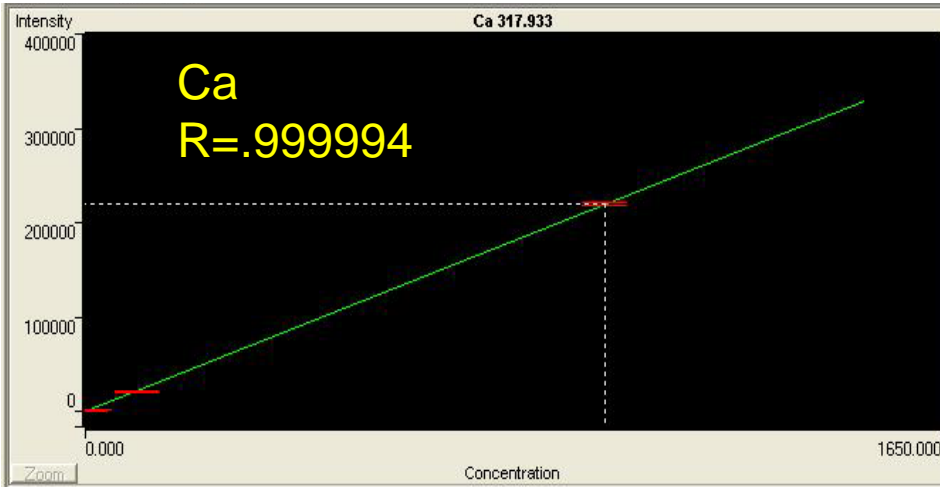
General settings

Replicates: 2

# Assist instrument method



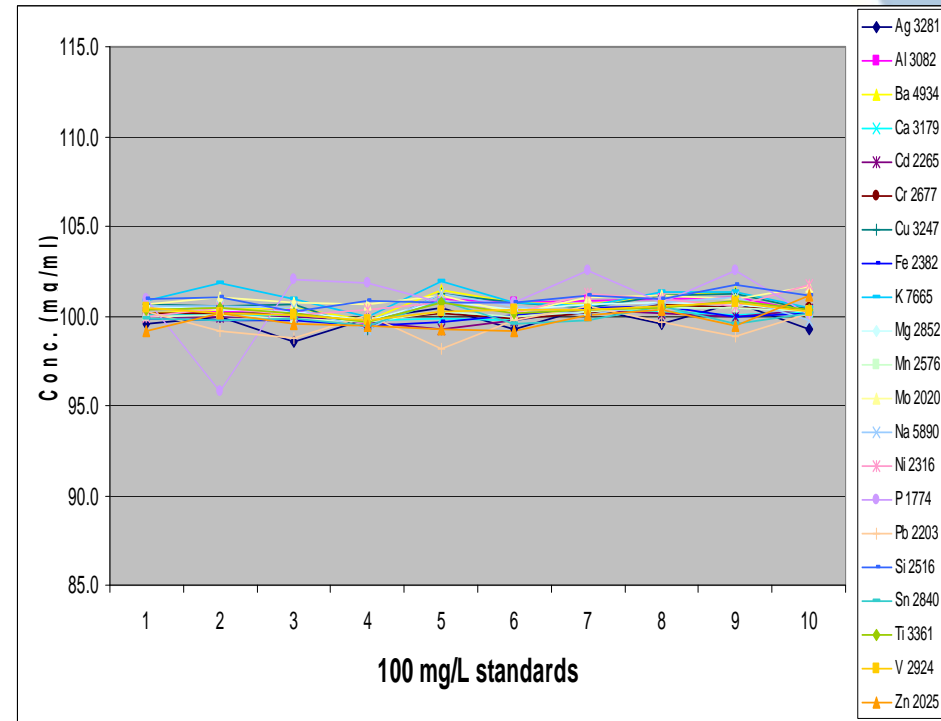
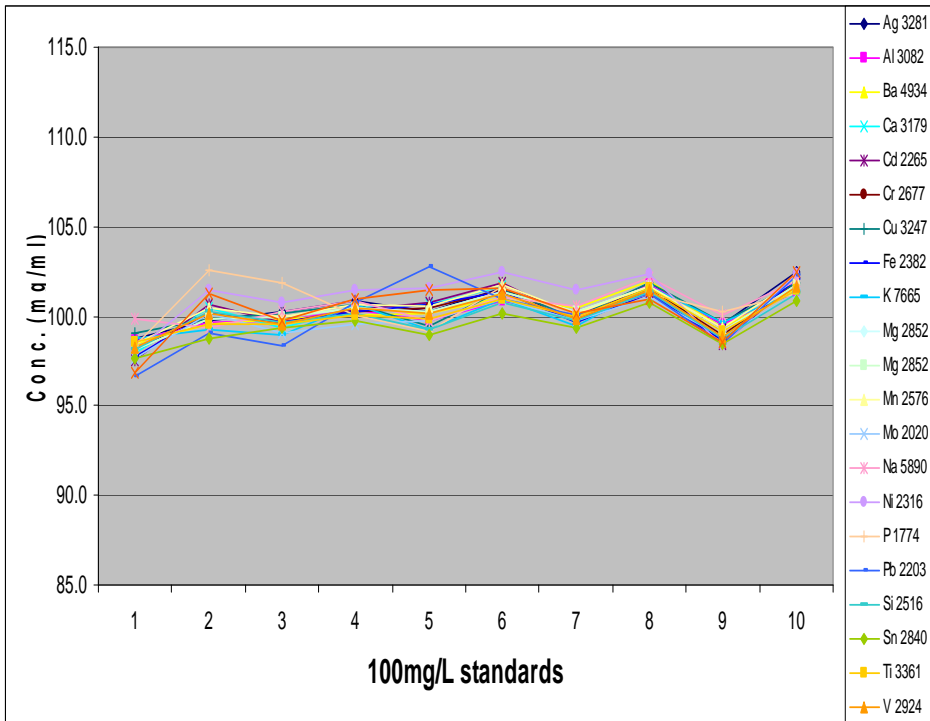
# Assist calibration



# Reproducibility comparison

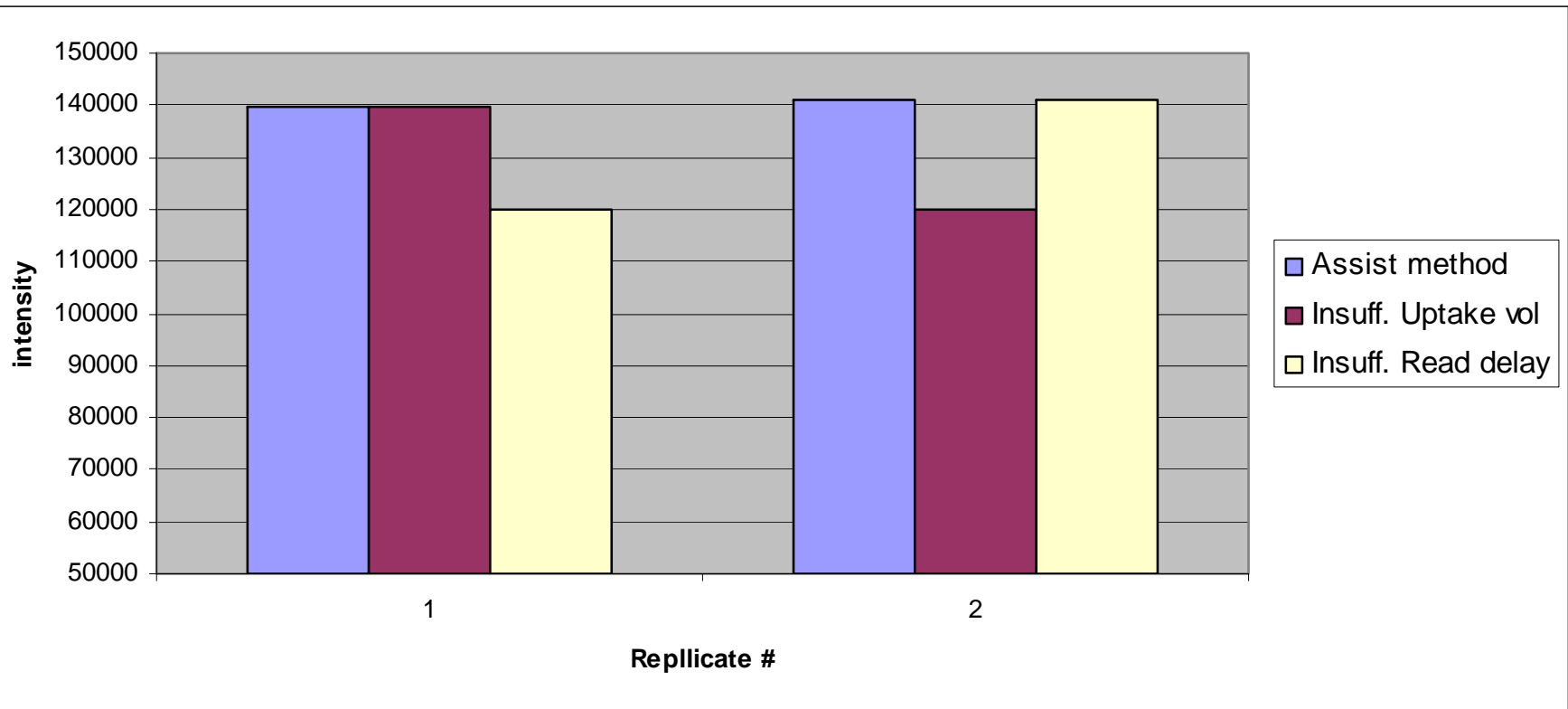
Undiluted oil without rinse

Diluted oil without rinse



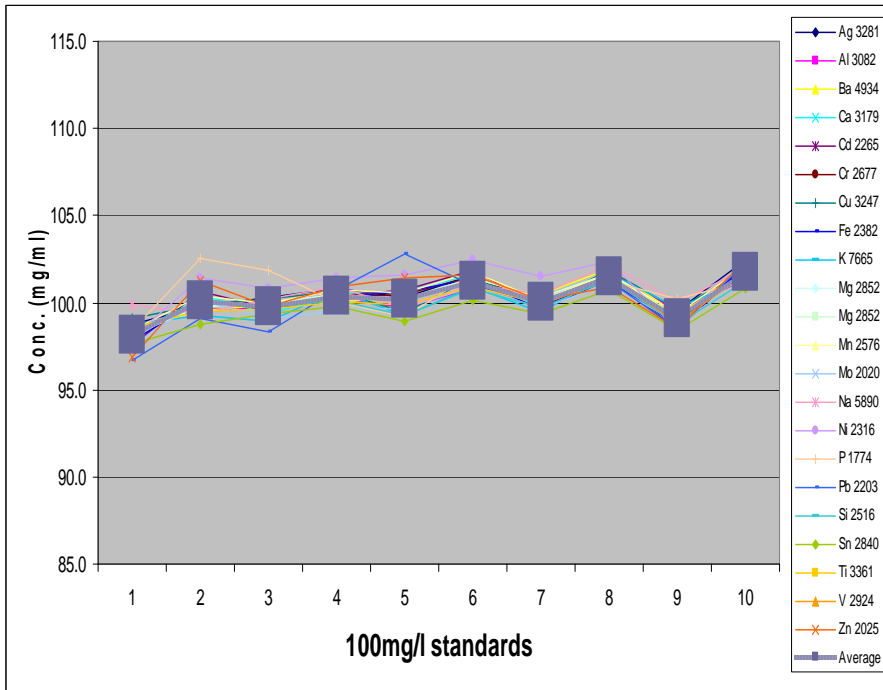
# Replicate study

Average of ten 100mg/L stds for 21 lines

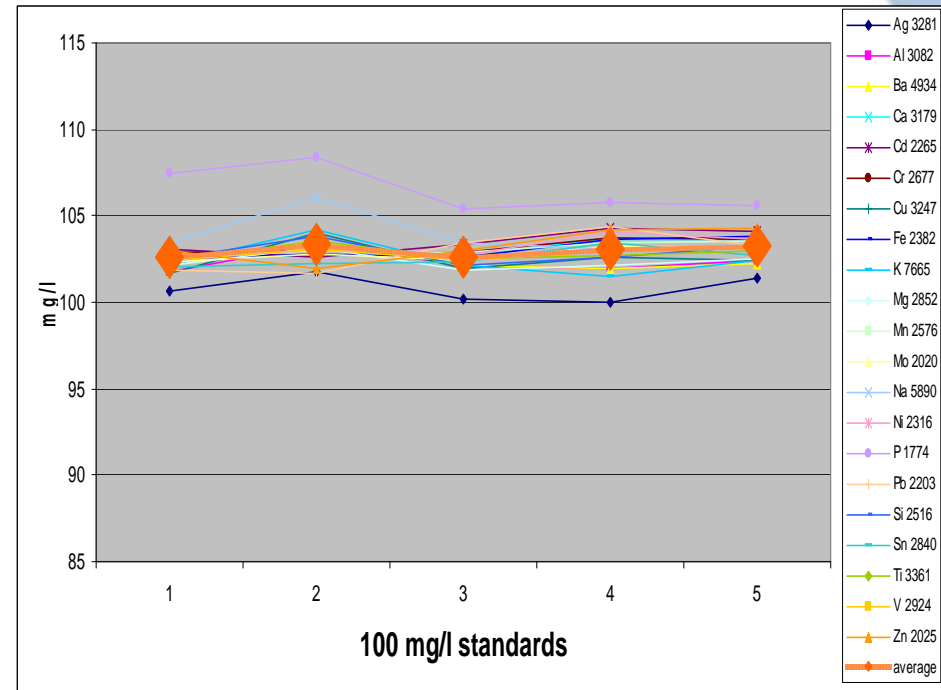


# Comparison of dilution accuracy

Assist

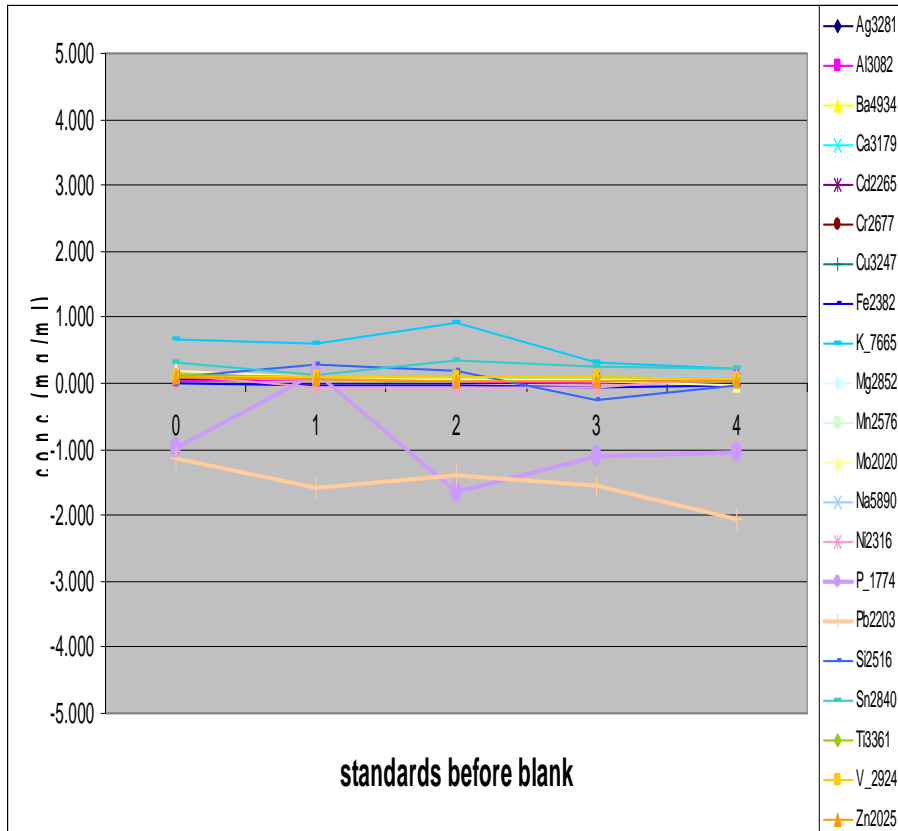


Niagara Plus after manual dilution

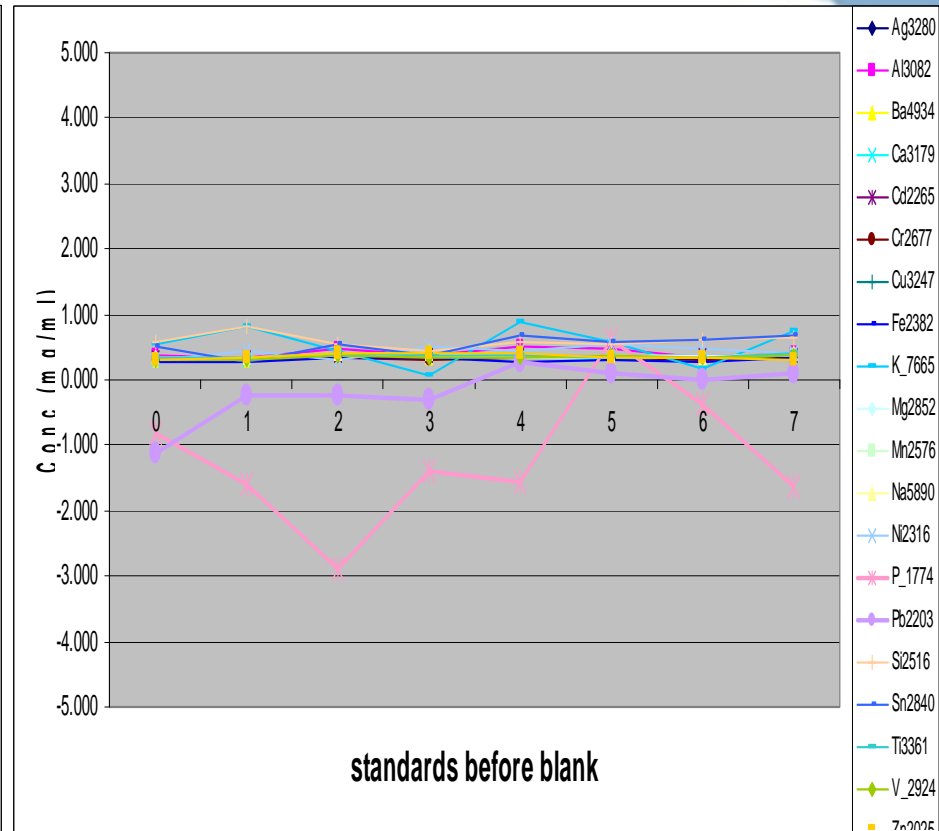


# Carryover comparison

Undiluted oil without rinse (Assist)



Diluted oil without rinse (Niagara Plus)



# Summary

## Niagara Plus

- Requires off-line dilution
- RSD's <1.0%
- No rinse
  - 18.5s/sample
  - <0.3% carryover
- 1s rinse
  - 25s/sample
  - <0.2% carryover

## Assist

- Uses raw undiluted oil
- RSD's <3%
- No rinse
  - 28s/sample
  - <0.2% carryover