1999

Nitrate Analysis

Nilton N. Cometti

NITRATE ANALYSIS

Nilton Nélio Cometti

REASONS FOR ANALYZING NITRATE

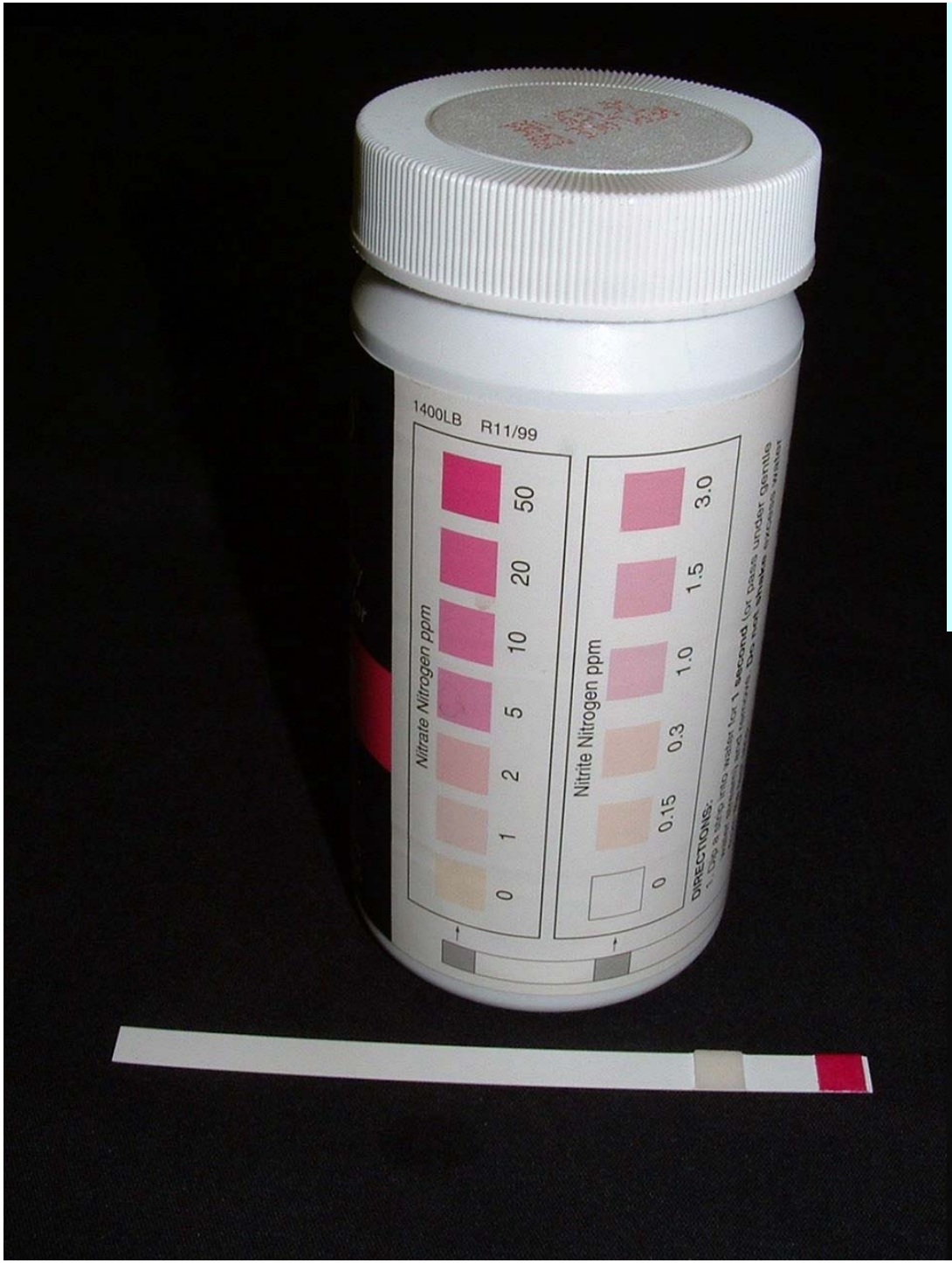
- NO_3^- is a major nutrient;
- Vegetable NO_3^- accumulation;
- Hazardous NO_3^-;
- Ground water NO_3^- pollution;
- NO_3^- is a plant nutrition status indicator.

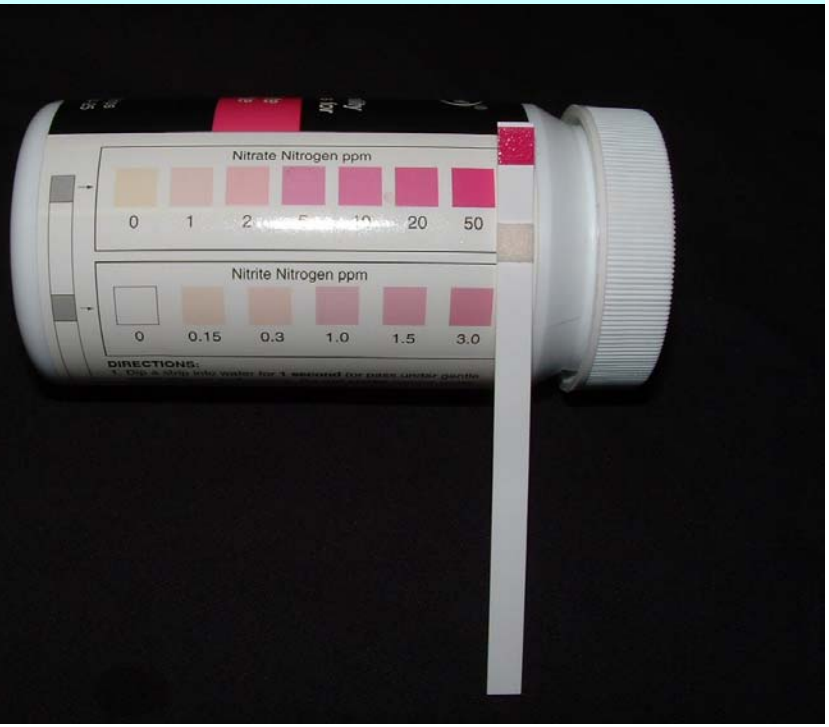
OBJECTIVE:

DEVELOP A SUITABLE, FAST, AND RELIABLE METHOD OF NITRATE ANALYSIS IN PLANT TISSUES

NITRATE ANALYSIS METHODS

NITRATE STRIP TEST



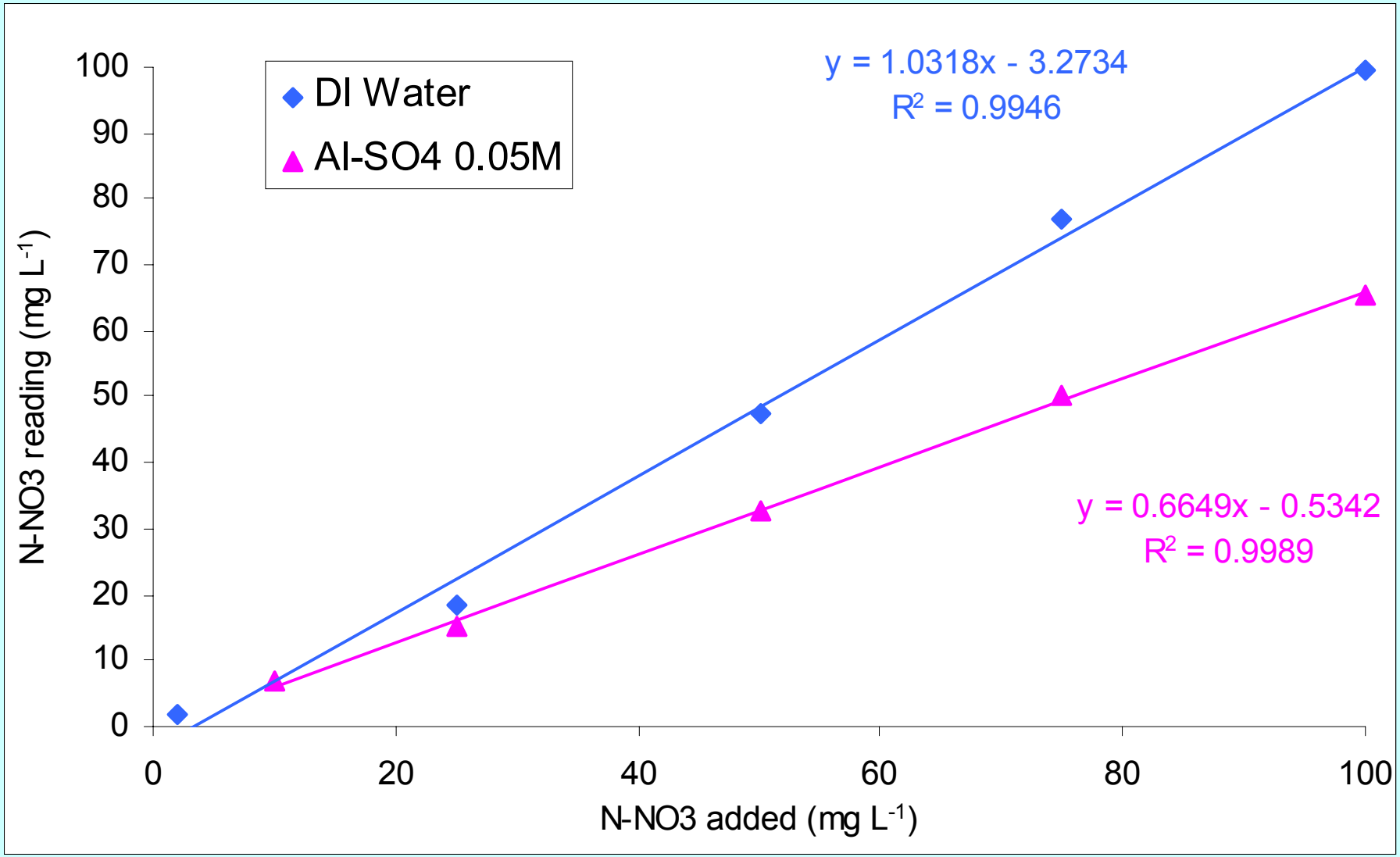



REPLACE CAP
AFTER USE
Aquachek
HACH
Water Quality
Test Strips for
Nitrate
Nitrite
25 Test Strips
Cat. 27454-25

CARDY ION METER

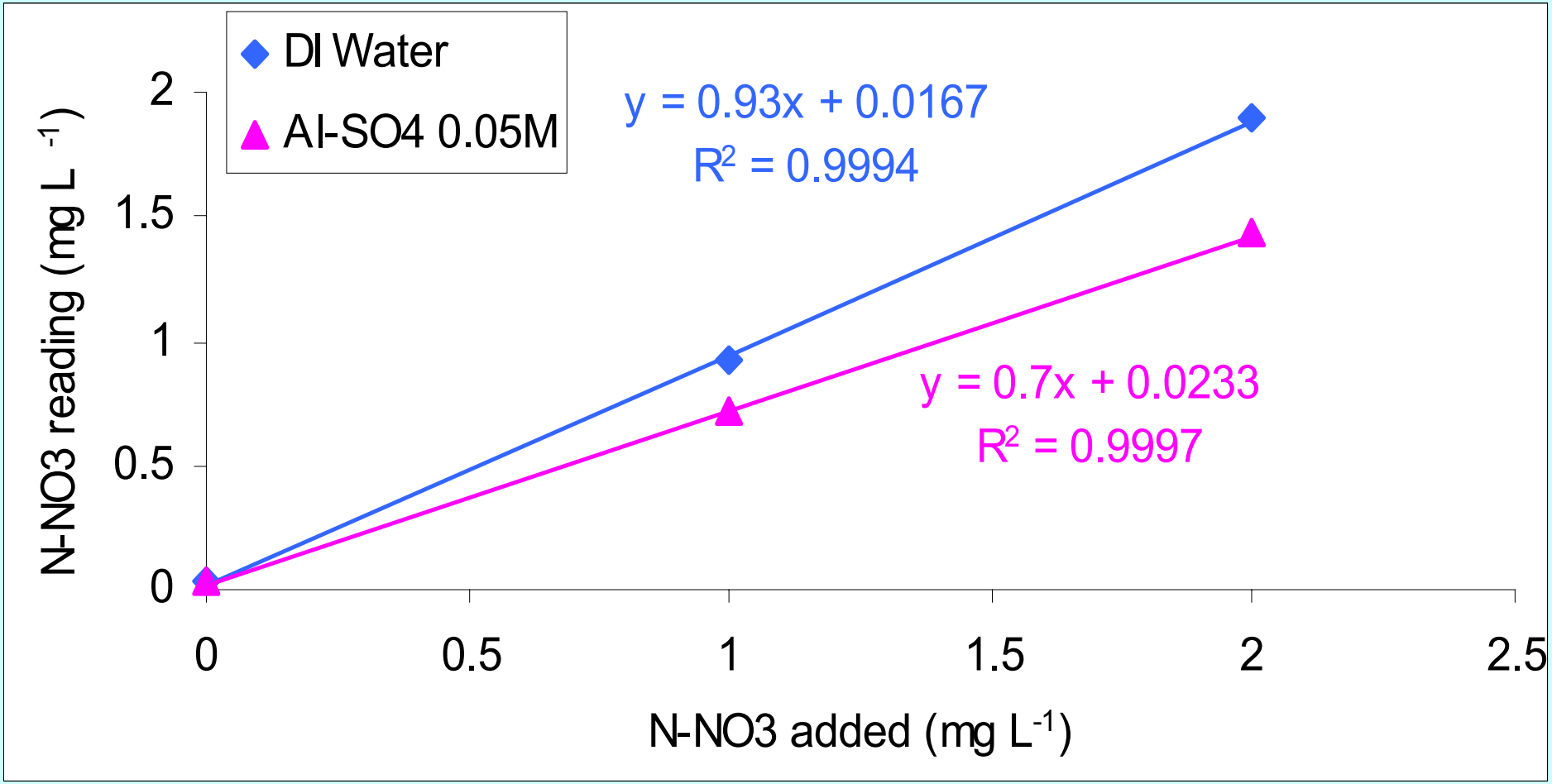
CARDY ION METER

CARDY ION METER CALIBRATION

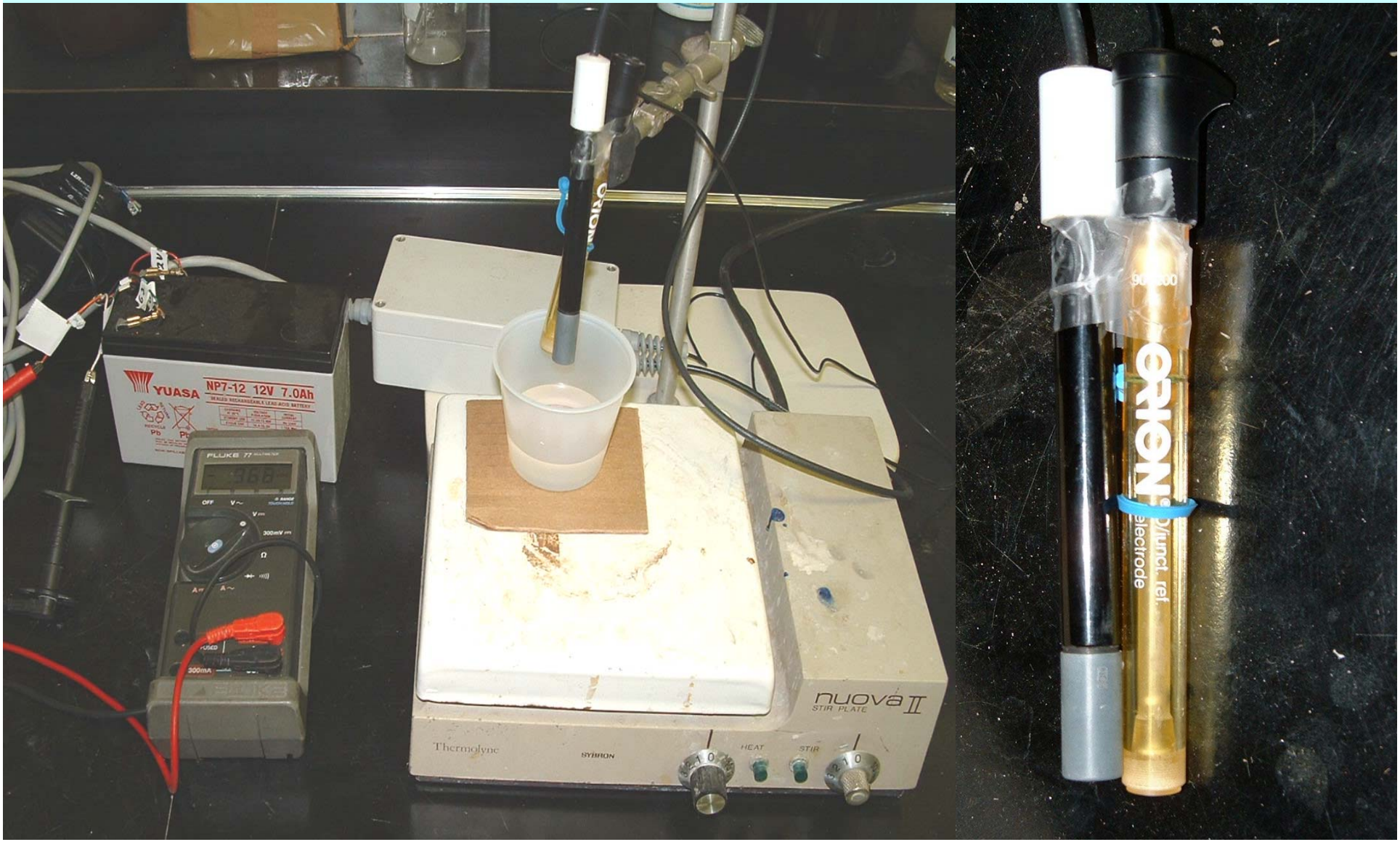


SMART COLORIMETER

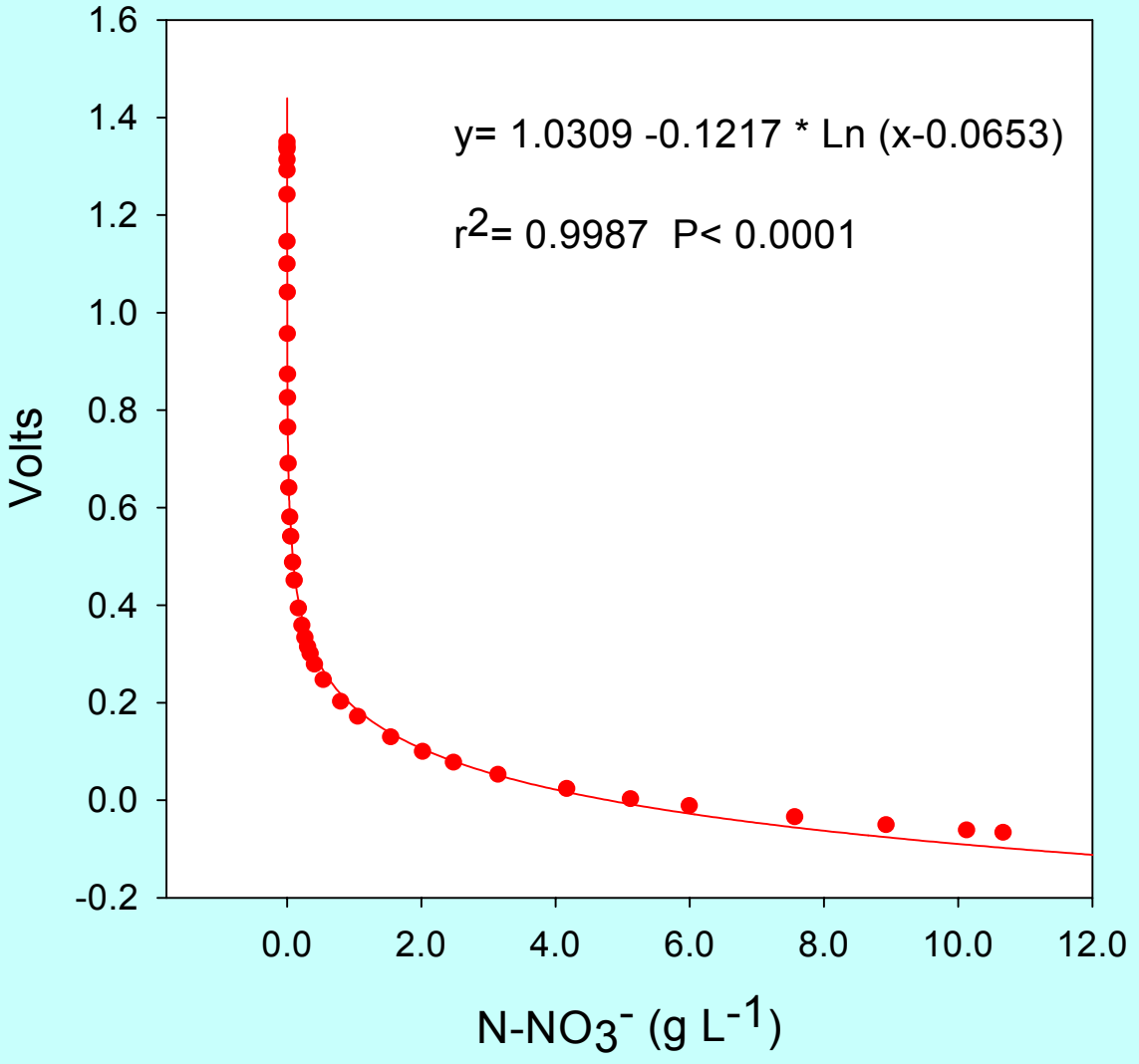
SMART COLORIMETER CALIBRATION



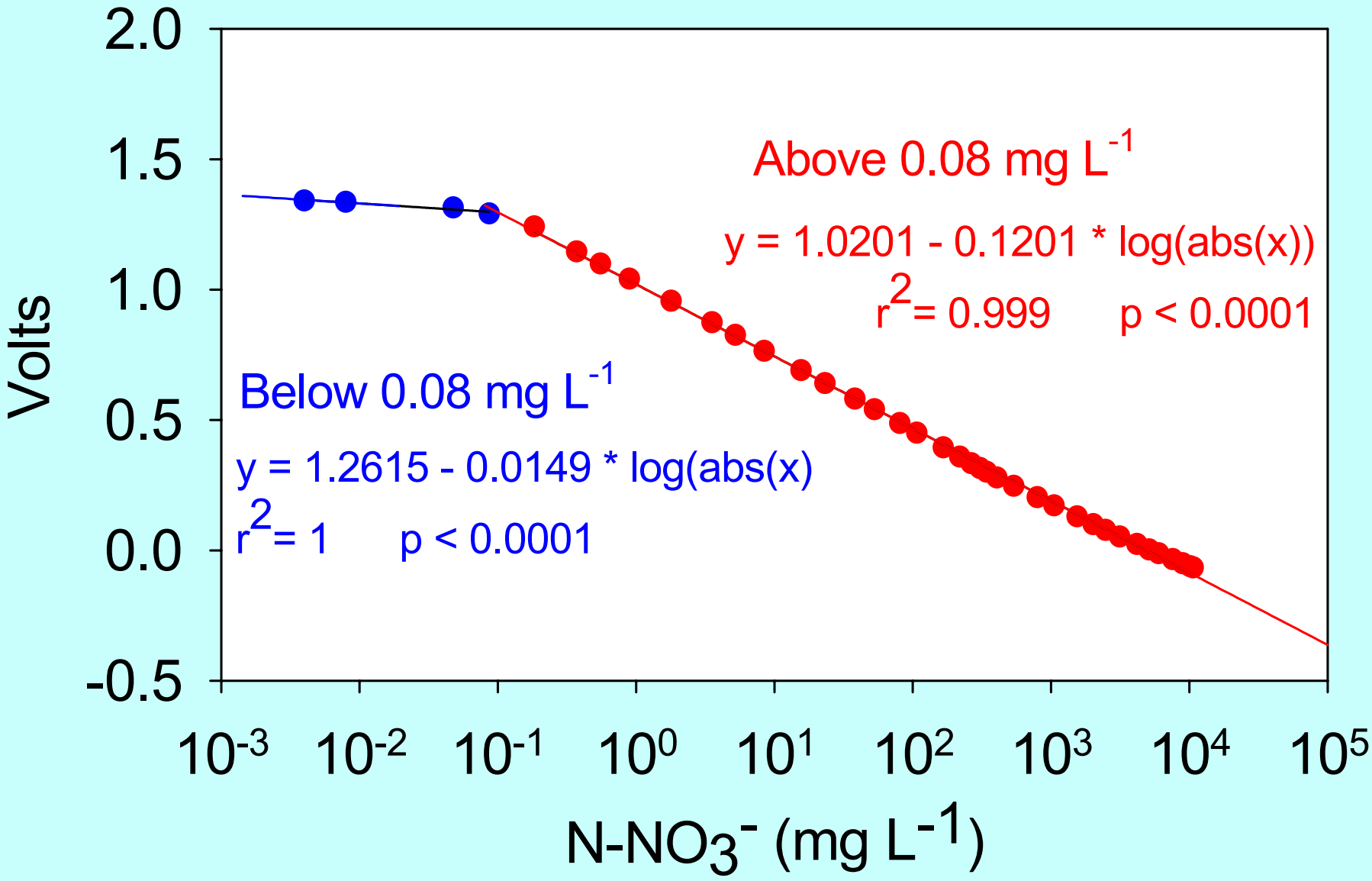
NITRATE SELECTIVE ELECTRODE



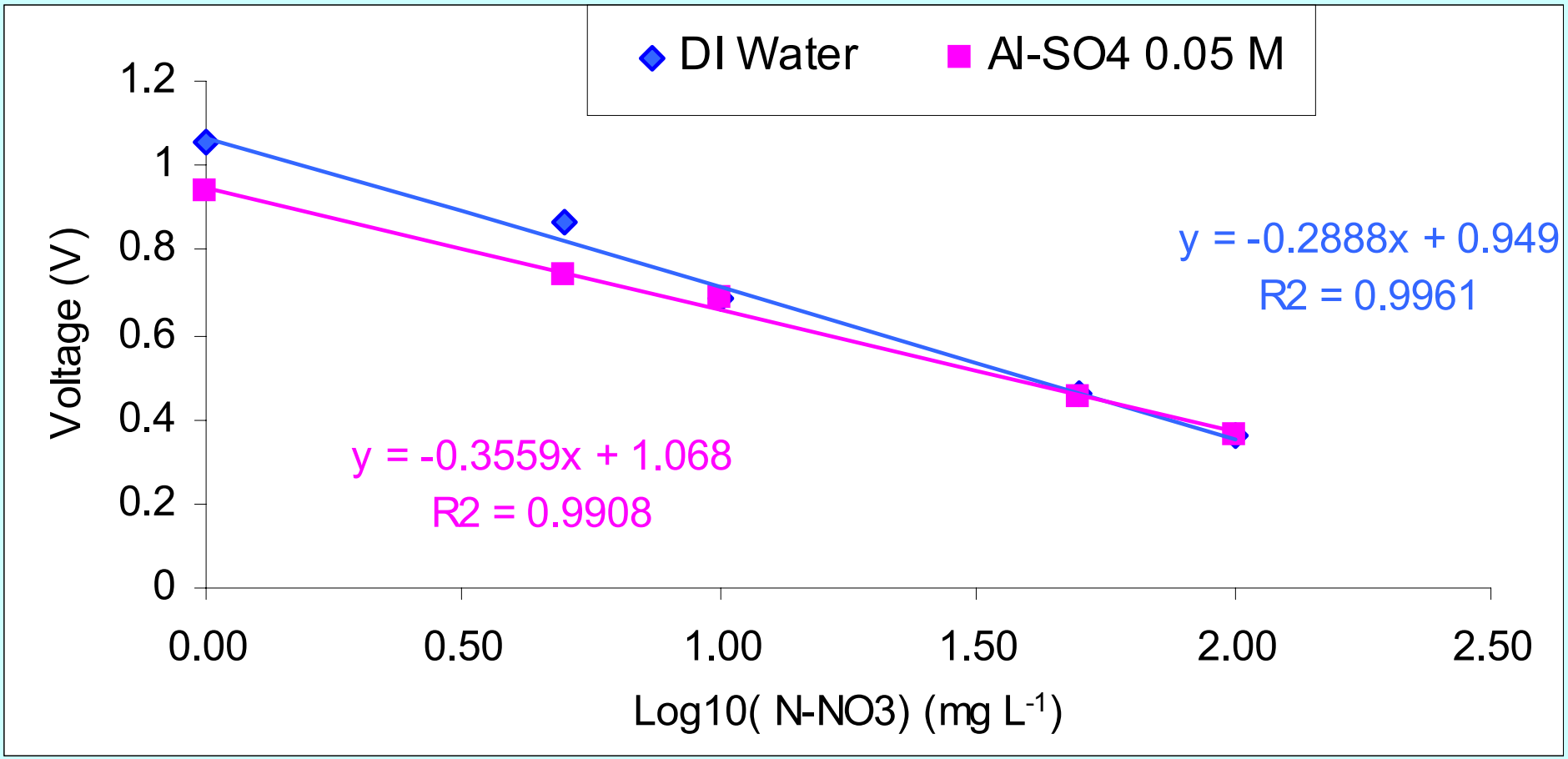
NITRATE ELECTRODE RESPONSE



NITRATE ELECTRODE RESPONSE

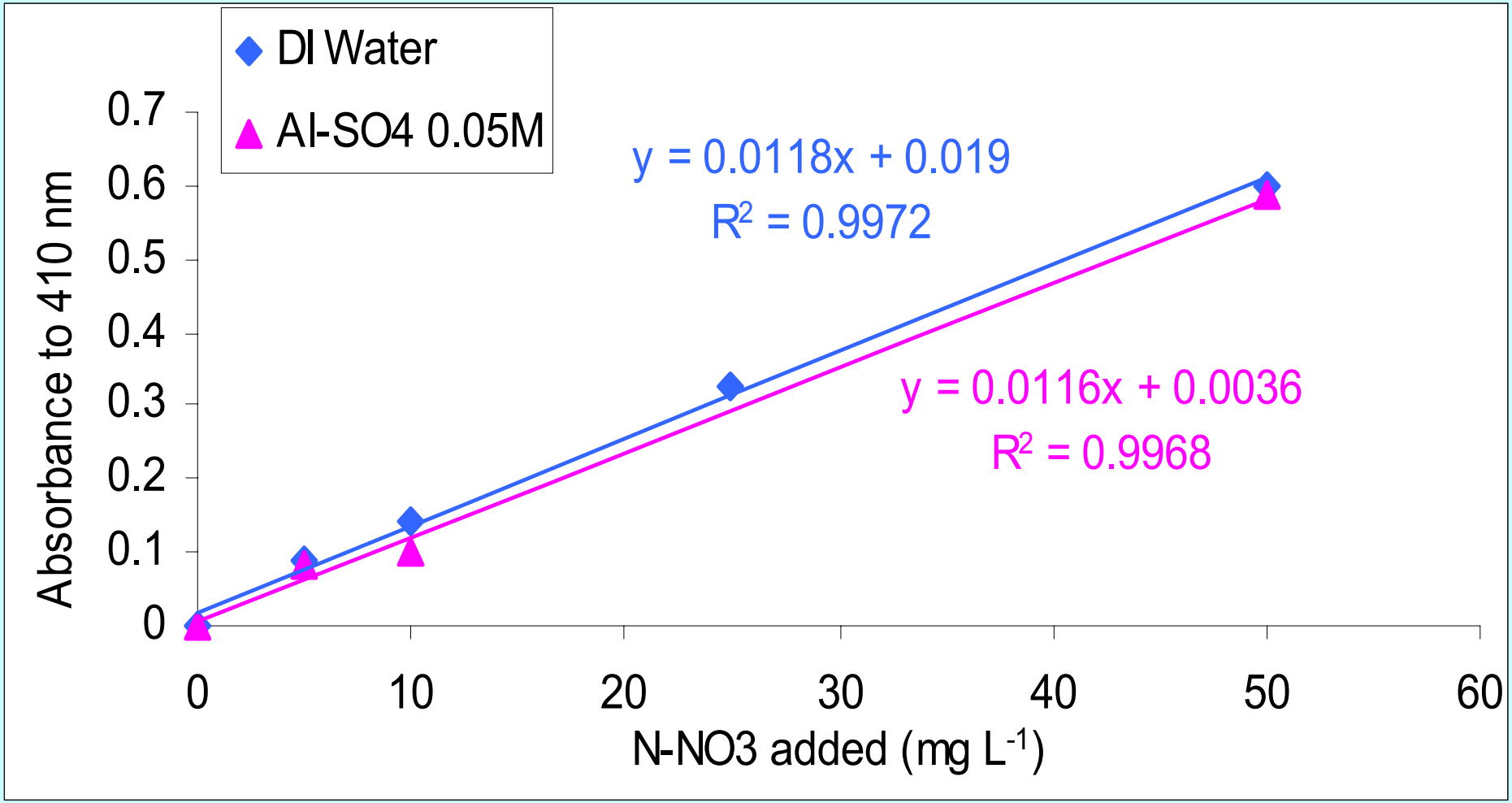


NITRATE ELECTRODE CALIBRATION



SALICYLIC ACID

SALICYLIC ACID CALIBRATION



FIRST TRIAL – MATERIAL AND METHODS

1- LETTUCE TISSUE

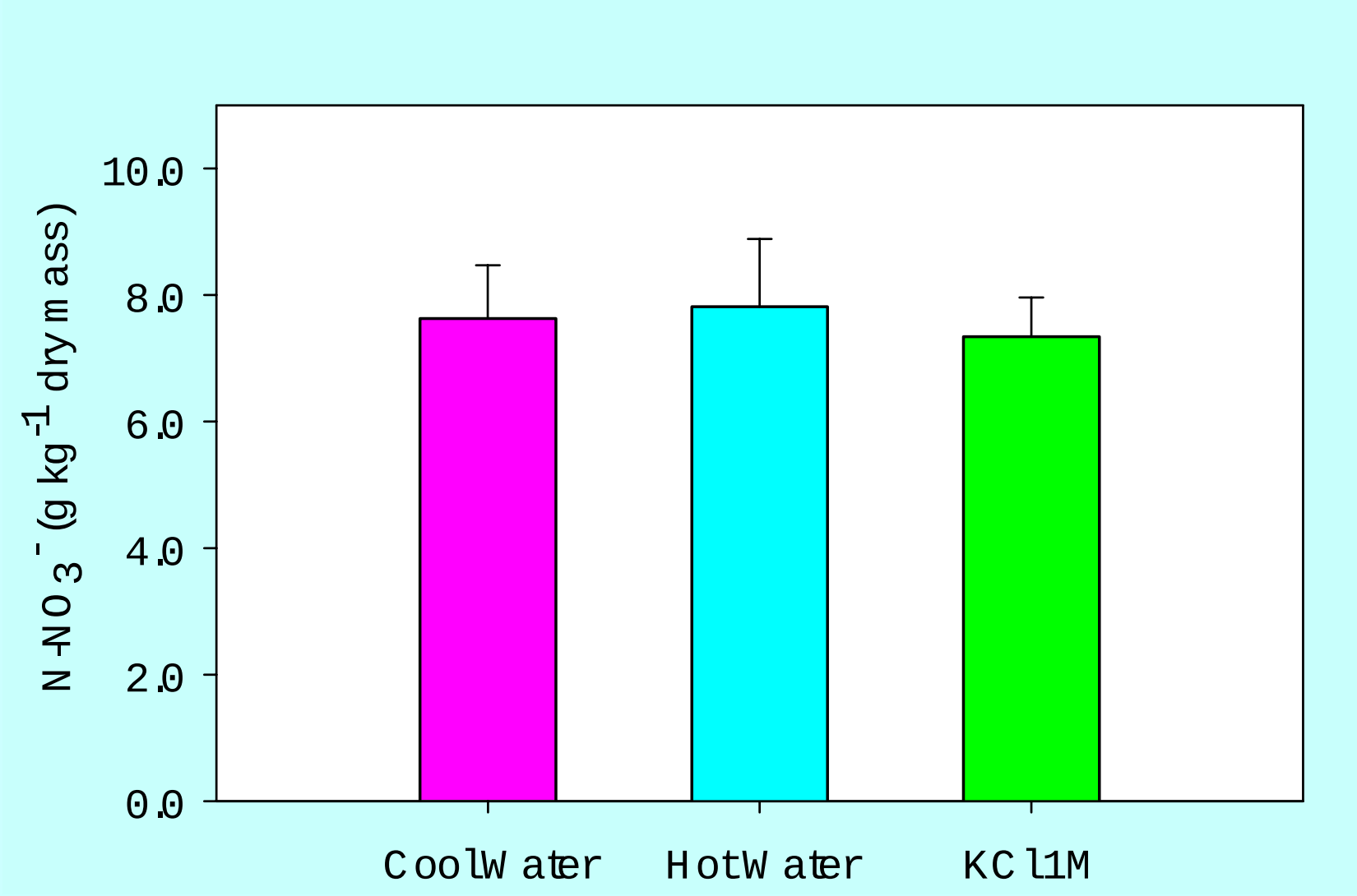
2- a) "SMART COLORIMETER" & NSE (NITRATE SELECTIVE ELECTRODE):

3- COLD WATER, HOT WATER, AND KCl 1 mol L^{-1};

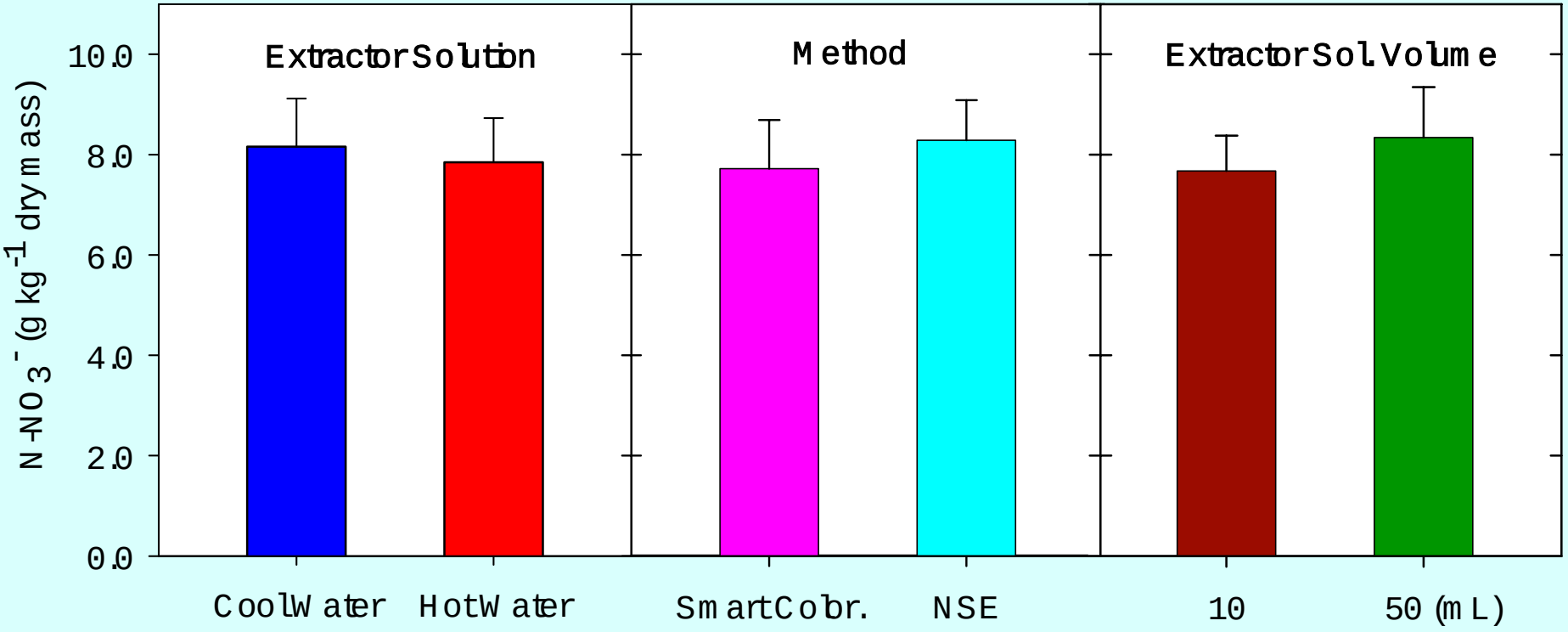
3- VOLUME OF SOLUTION: 10 AND 50 mL;

4- DIFFERENT MASS, SOLUTION VOLUME AND EXTRACTION TIME.

$N\text{-}NO_3^-$ in Lettuce Leaf Tissue: Extractor Solution Test with Smart Colorimeter Method



$N\text{-}NO_3^-$ in Lettuce Leaf Tissue: Extractor Solution, Method, and Extractor Sol. Volume Test.



SECOND TRIAL – MATERIAL AND METHODS

1- MATERIAL: FRESH AND DRIED RICE LEAF TISSUE FROM THREE NITRATE LEVELS IN THE NUTRIENT SOLUTION:

A) HIGH (~ 6 mmol L^{-1});

B) MEDIUM TO LOW (0.5 TO 0.1 mmol L^{-1});

C) LOW TO MEDIUM (0.1 TO 0.5 mmol L^{-1});

2- EXTRACTOR SOLUTIONS:

- DI WATER;
- $Al_2(SO_4)_3.18\ H_2O$ 0.05 mol L^{-1};

3- VOLUME OF SOLUTION: 50 mL;

4- SAMPLE WEIGHT: 2.0 AND 0.2 g OF FRESH AND DRIED MASS RESPECTIVELY;

SECOND TRIAL – MATERIAL AND METHODS

5- METHOD OF ANALYSIS:

A) NITRATE STRIP TEST

B) CARDY ION METER

C) SMART COLORIMETER

D) NSE (NITRATE SELECTIVE ELECTRODE)

E) SALYCILIC ACID

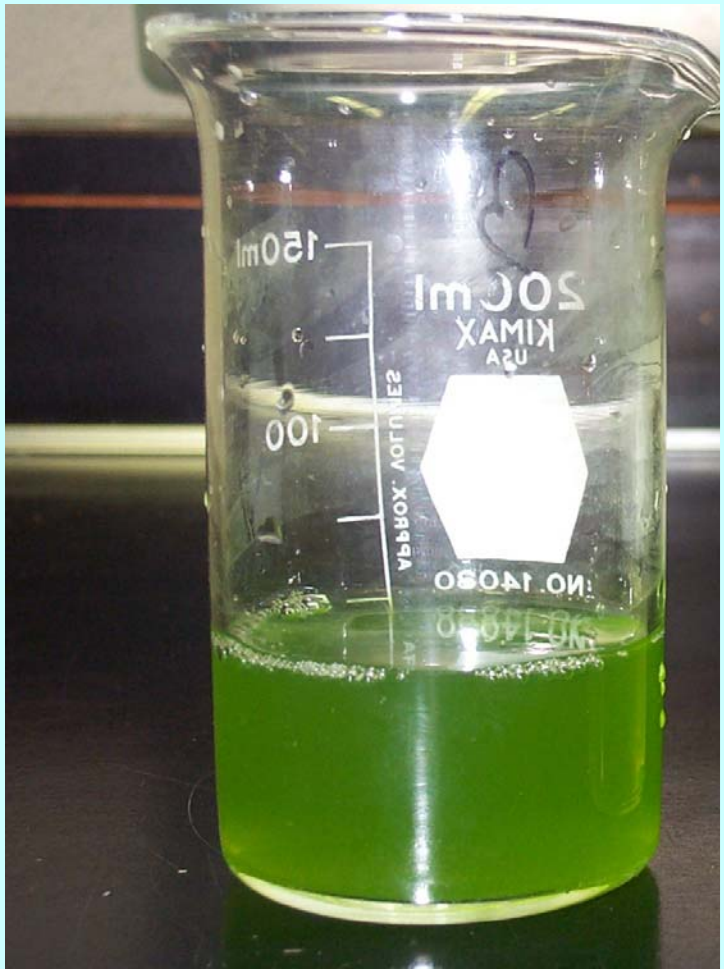
SECOND TRIAL – MATERIAL AND METHODS

6- SEQUENCE OF EXTRACTION AND READING:

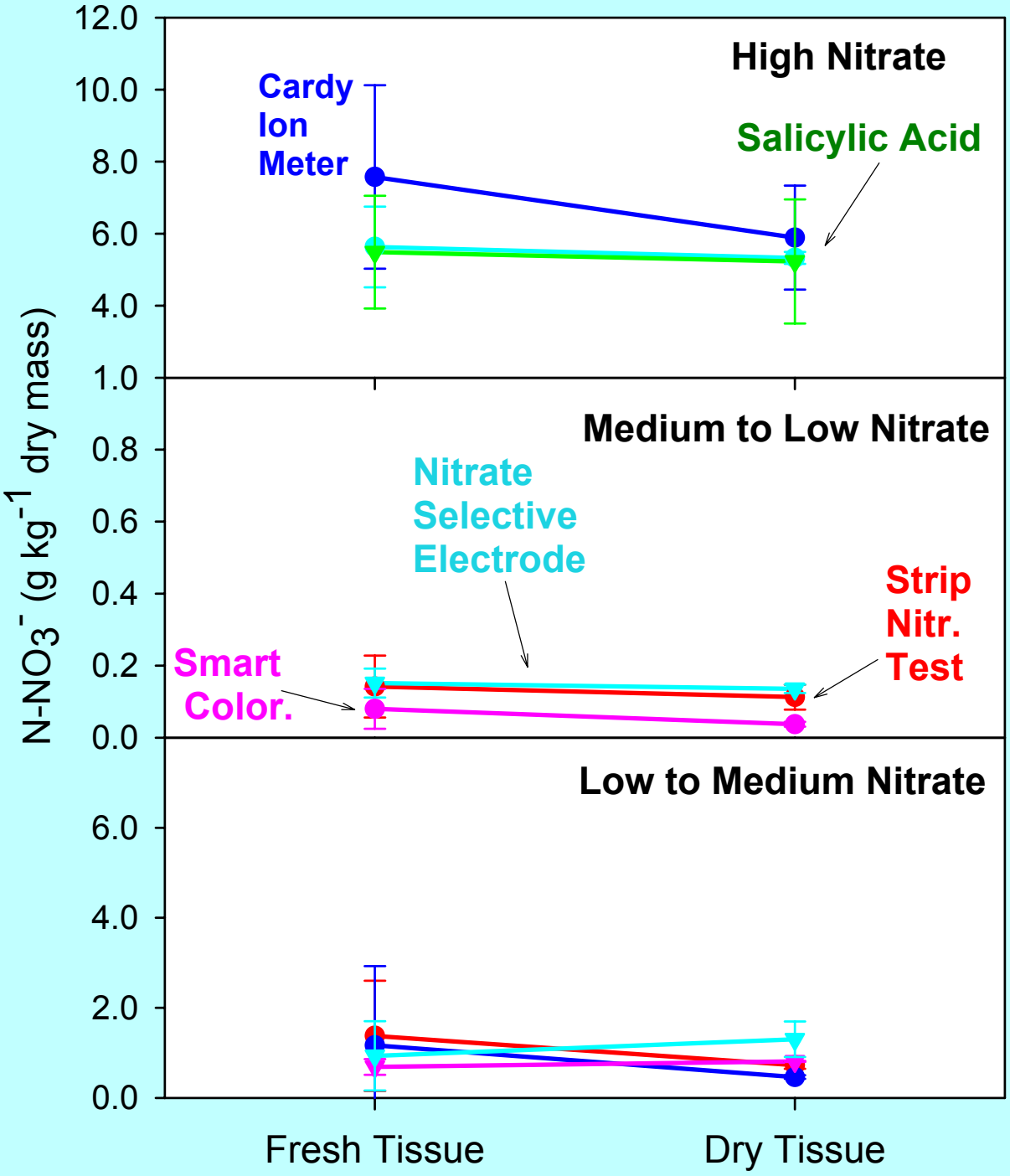
- WEIGHTING;
- EXTRACTOR SOLUTION ADDITION;
- BLENDER SHAKING DURING 20 SECONDS FOR FRESH SAMPLES;
- MANUALY SHAKING FOR 10 SECONDS THREE TIMES WITHIN ONE HOUR FOR DRIED SAMPLES;
- DILUTION WHEN RECESSARY;
- N-NO3 READINGS;
- FILTRATION WITH WATMAN 4;
- SECOND READINGS;
- CHARCOAL ADDITION (0.5 g) ONLY ON FRESH SAMPLES;
- SECOND FILTRATION;
- THIRD READINGS;

EXTRACT AFTER FILTRATION

DI WATER

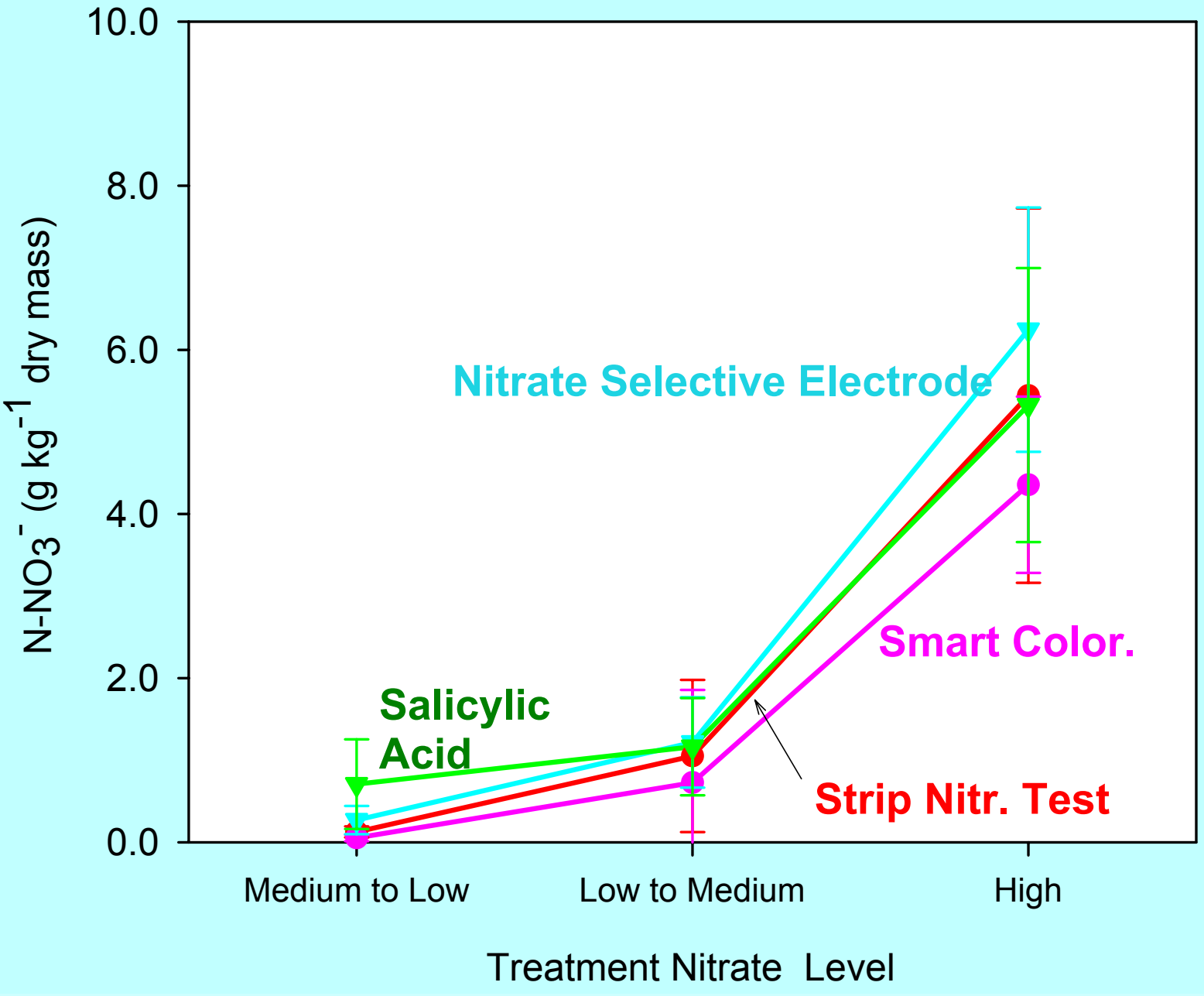


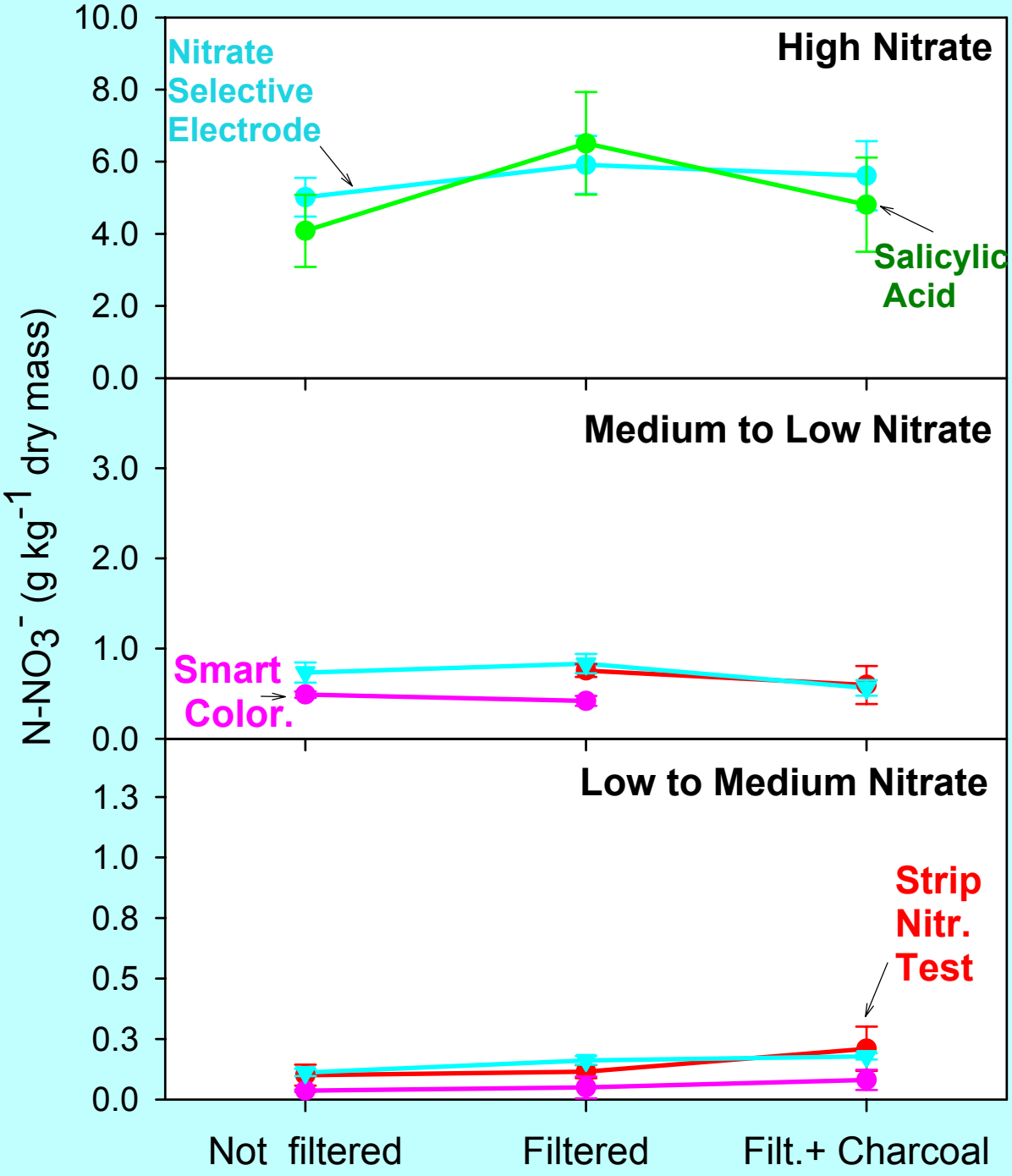
Al-SO_4 0.05 M


12.0
10.0
8.0
6.0
4.0
1.0
0.8
0.6
0.4
0.2
0.0
6.0
4.0
2.0
0.0
N-NO3- (g kg-1 dry mass)
High Nitrate
Cardy Ion Meter
Salicylic Acid
Medium to Low Nitrate
Nitrate Selective Electrode
Strip Nitr. Test
Smart Color.
Low to Medium Nitrate
Fresh Tissue
Dry Tissue

RICE TISSUE

FRESH AND DRY RICE TISSUE




High Nitrate
Nitrate Selective Electrode
Salicylic Acid
Medium to Low Nitrate
Smart Color.
Low to Medium Nitrate
Strip Nitr. Test
N-NO3- (g kg-1 dry mass)
10.0
8.0
6.0
4.0
2.0
0.0
3.0
2.0
1.0
0.0
1.3
1.0
0.8
0.5
0.3
0.0
Not filtered
Filtered
Filt.+ Charcoal

RICE TISSUE

THIRD TRIAL SETS

1- ONLY ONE EXTRACTOR SOLUTION: Al-SO_4 0.05 mol L^{-1}

2- LETTUCE GROWN HYDROPONICALLY WITH TWO NITRATE SOLUTION LEVELS: HIGH AND LOW.

3- NITRATE ANALYSIS METHODS:

A) NITRATE STRIP TEST

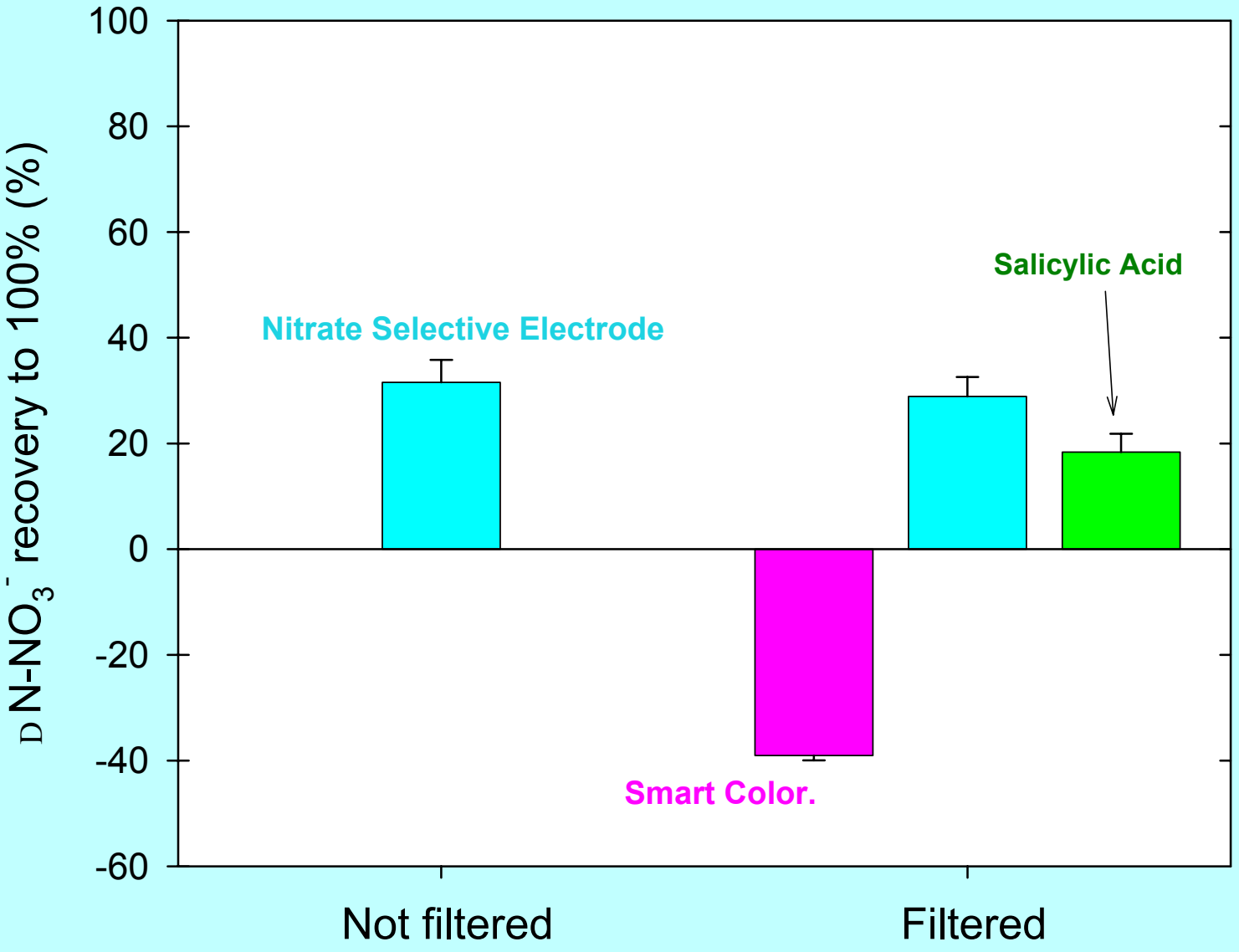
B) SMART COLORIMETER

C) NSE (NITRATE SELECTIVE ELECTRODE)

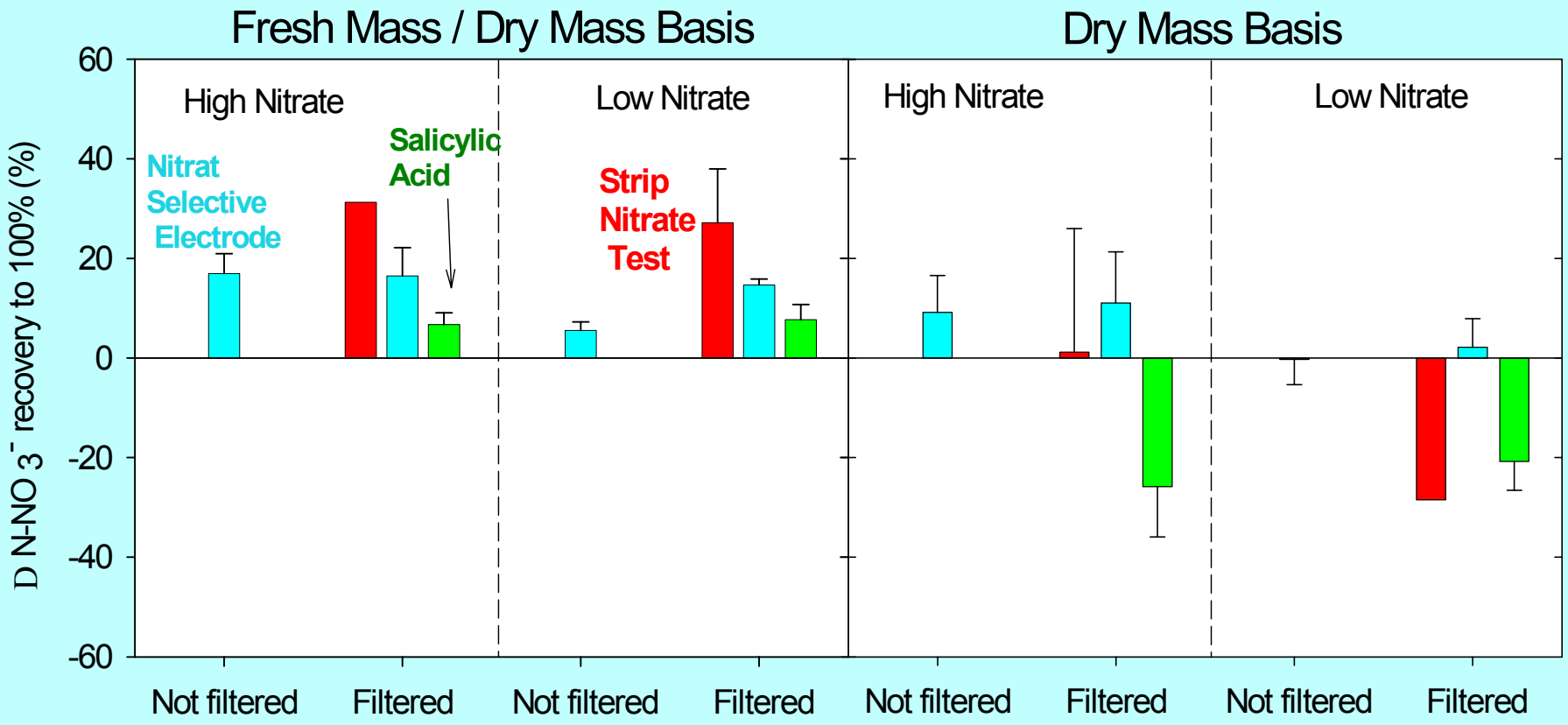
D) SALYCILIC ACID

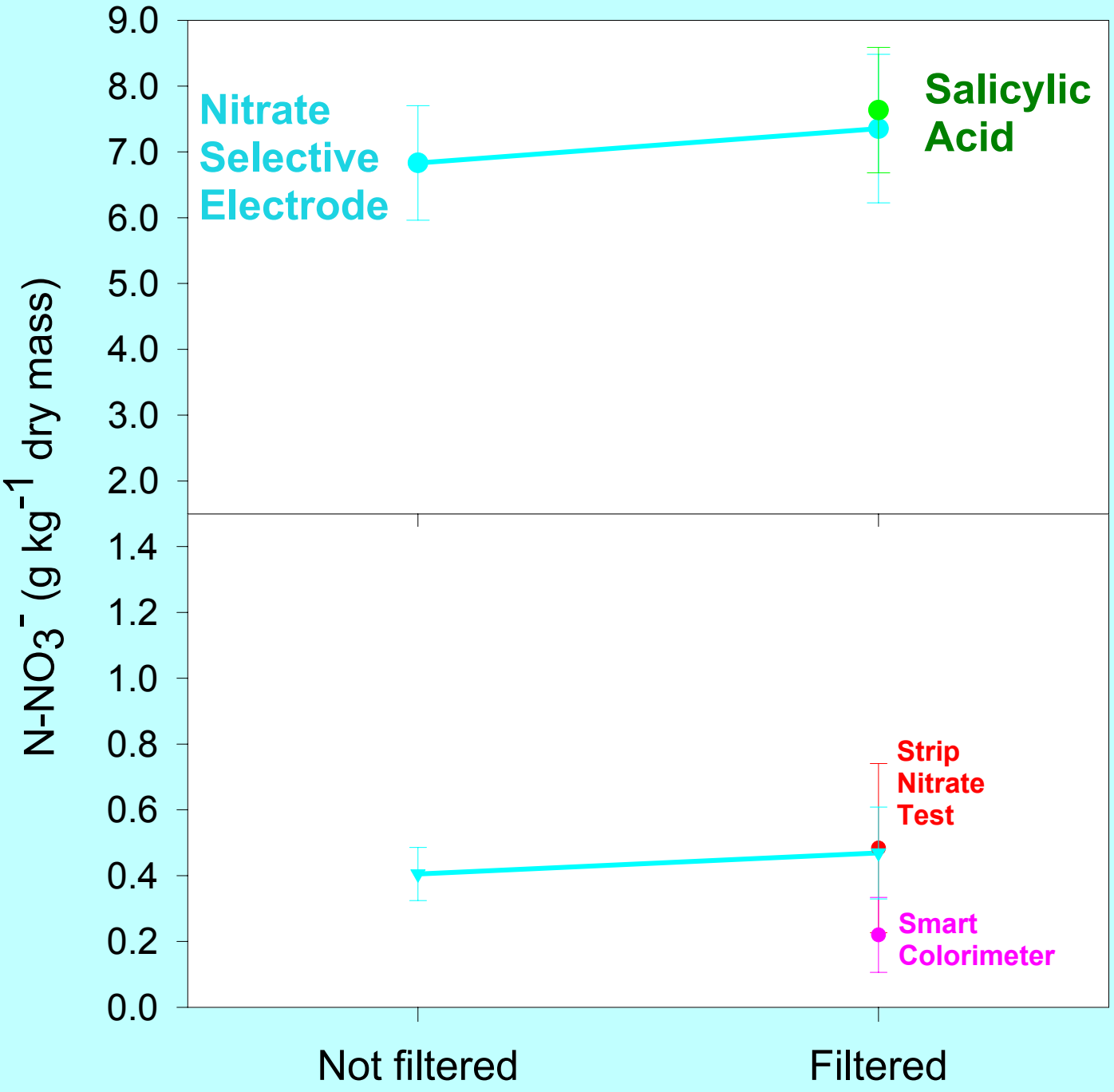
4- FILTRATION: NOT FILTERED AND FILTERED EXTRACTS.

NITRATE RECOVERY IN Al-SO4 EXTRACTOR SOLUTION

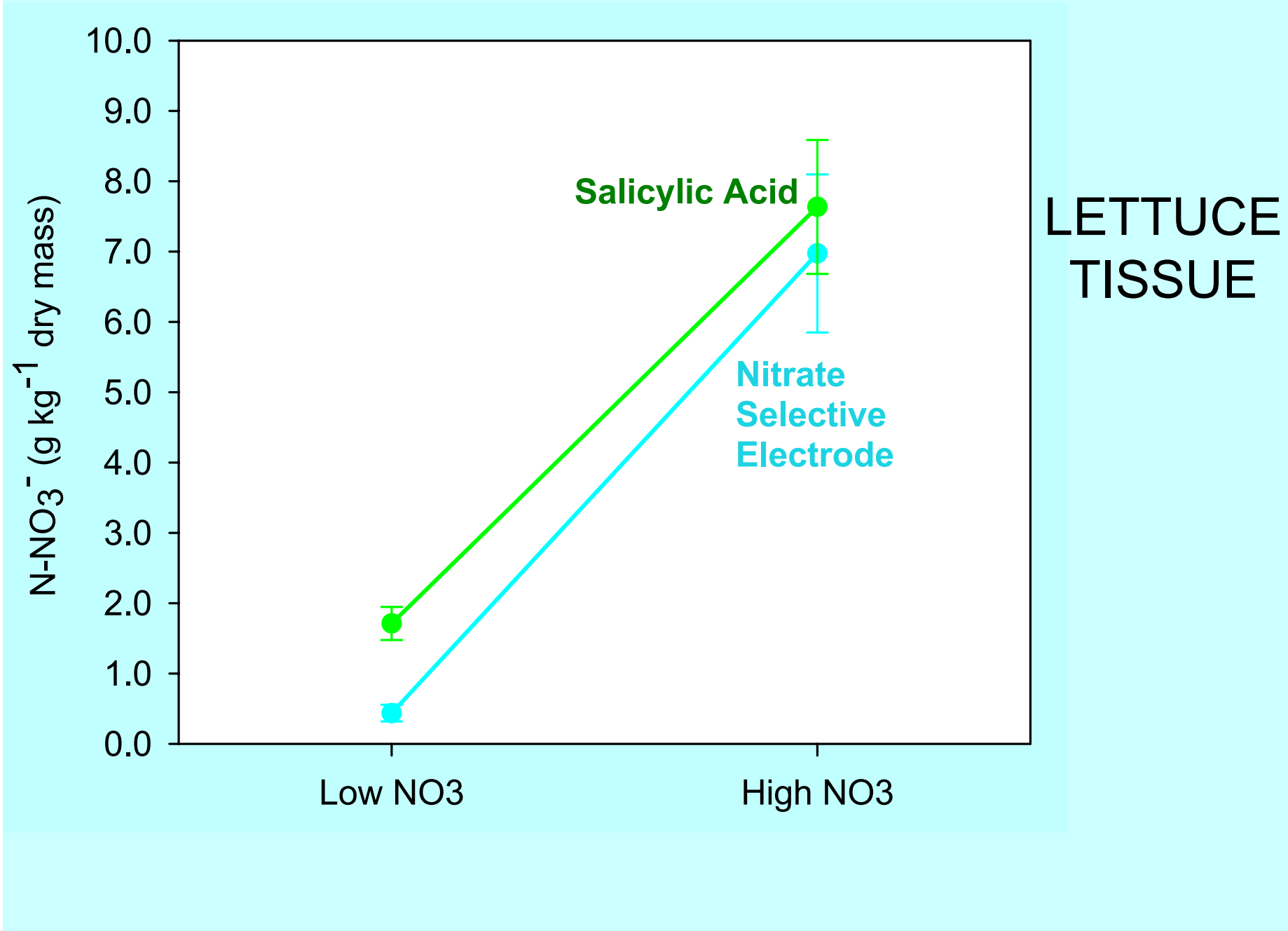


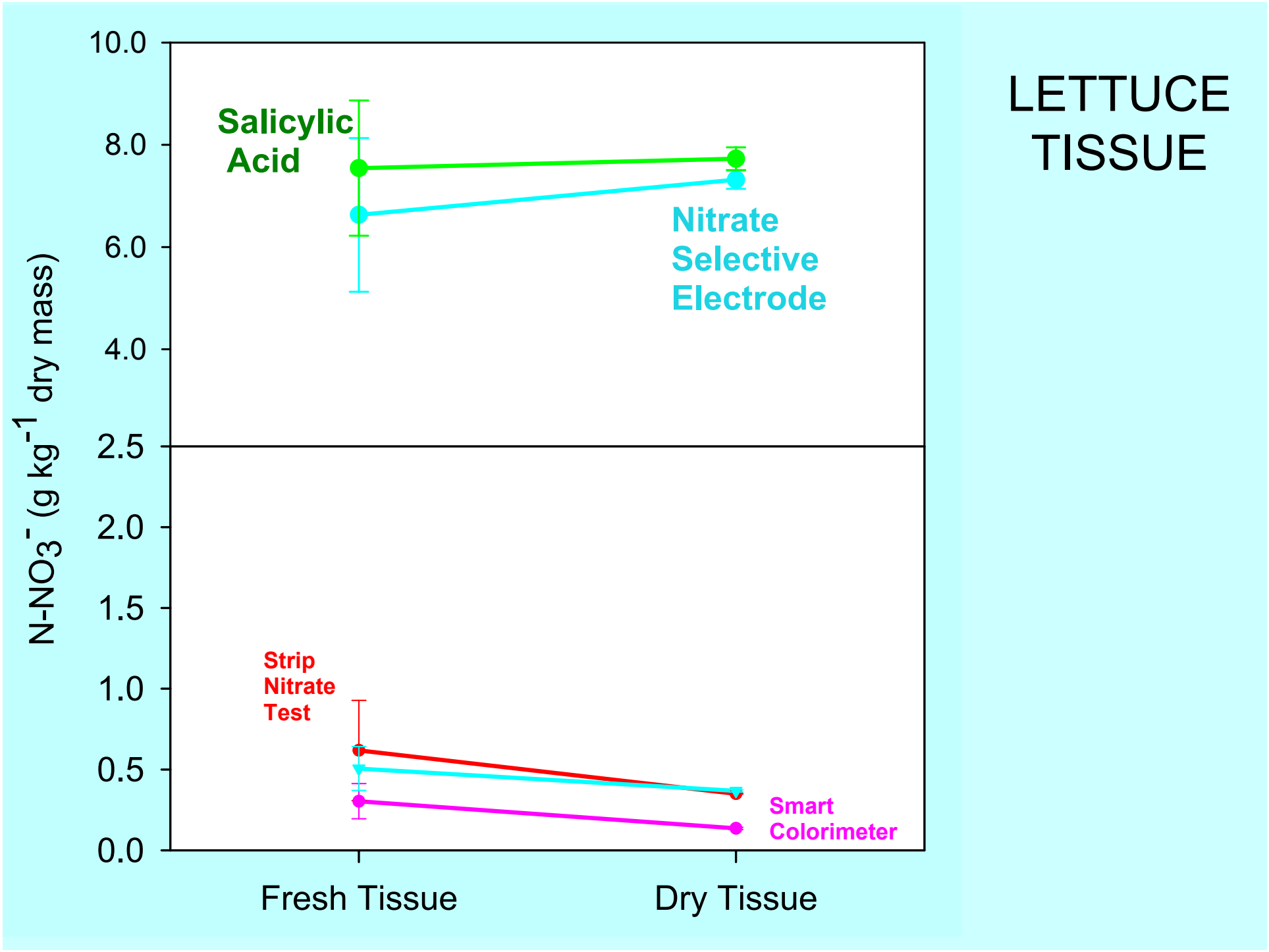
NITRATE RECOVERY IN LETTUCE TISSUE WHIT Al-SO4 EXTRACTOR SOLUTION





LETTUCE TISSUE

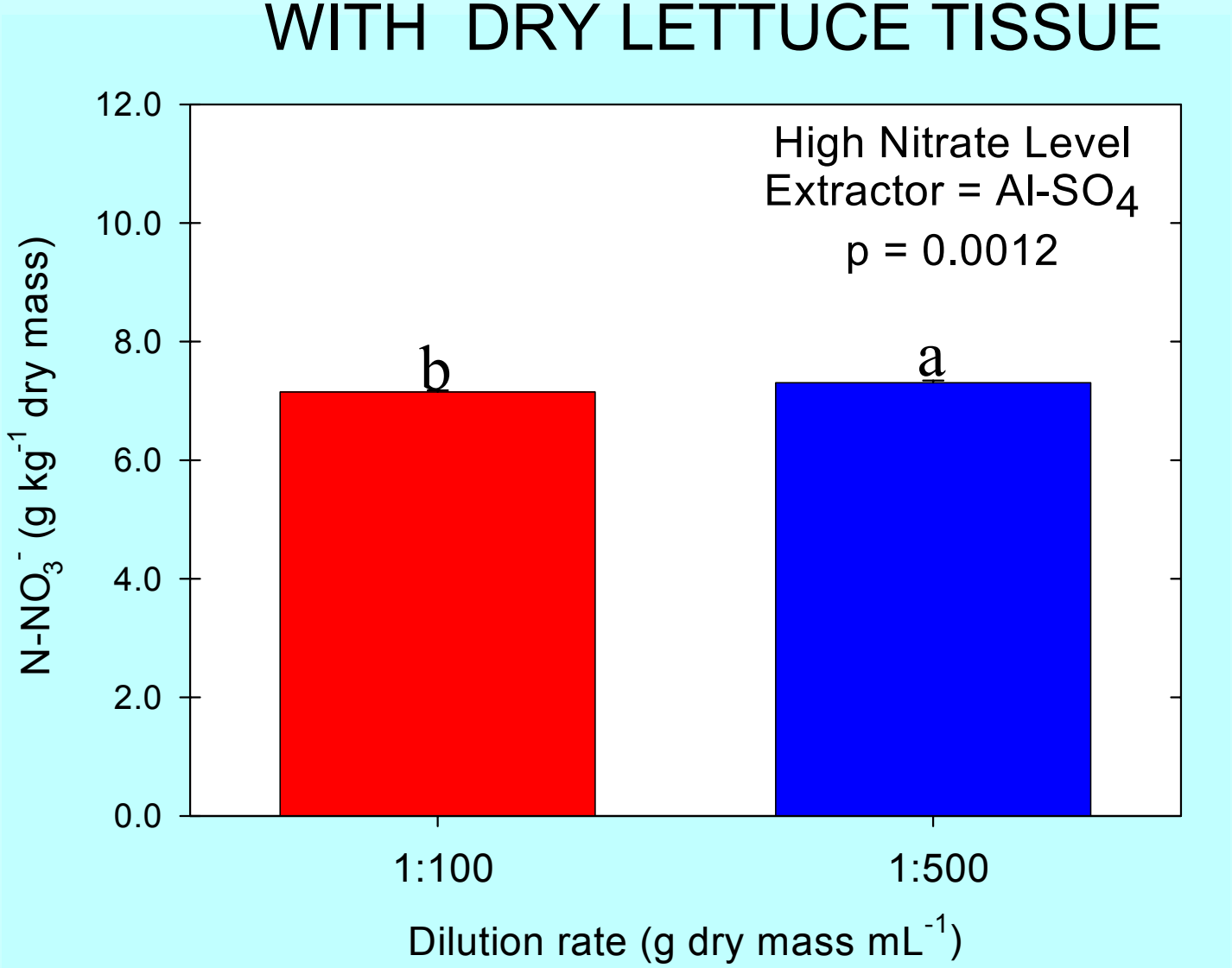

10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
N-NO3- (g kg-1 dry mass)
Salicylic Acid
Nitrate
Selective
Electrode
Low NO3
High NO3
LETTUCE
TISSUE


LETTUCE TISSUE
10.0
8.0
6.0
4.0
2.5
2.0
1.5
1.0
0.5
0.0
N-NO3- (g kg-1 dry mass)
Salicylic Acid
Nitrate Selective Electrode
Strip Nitrate Test
Smart Colorimeter
Fresh Tissue
Dry Tissue

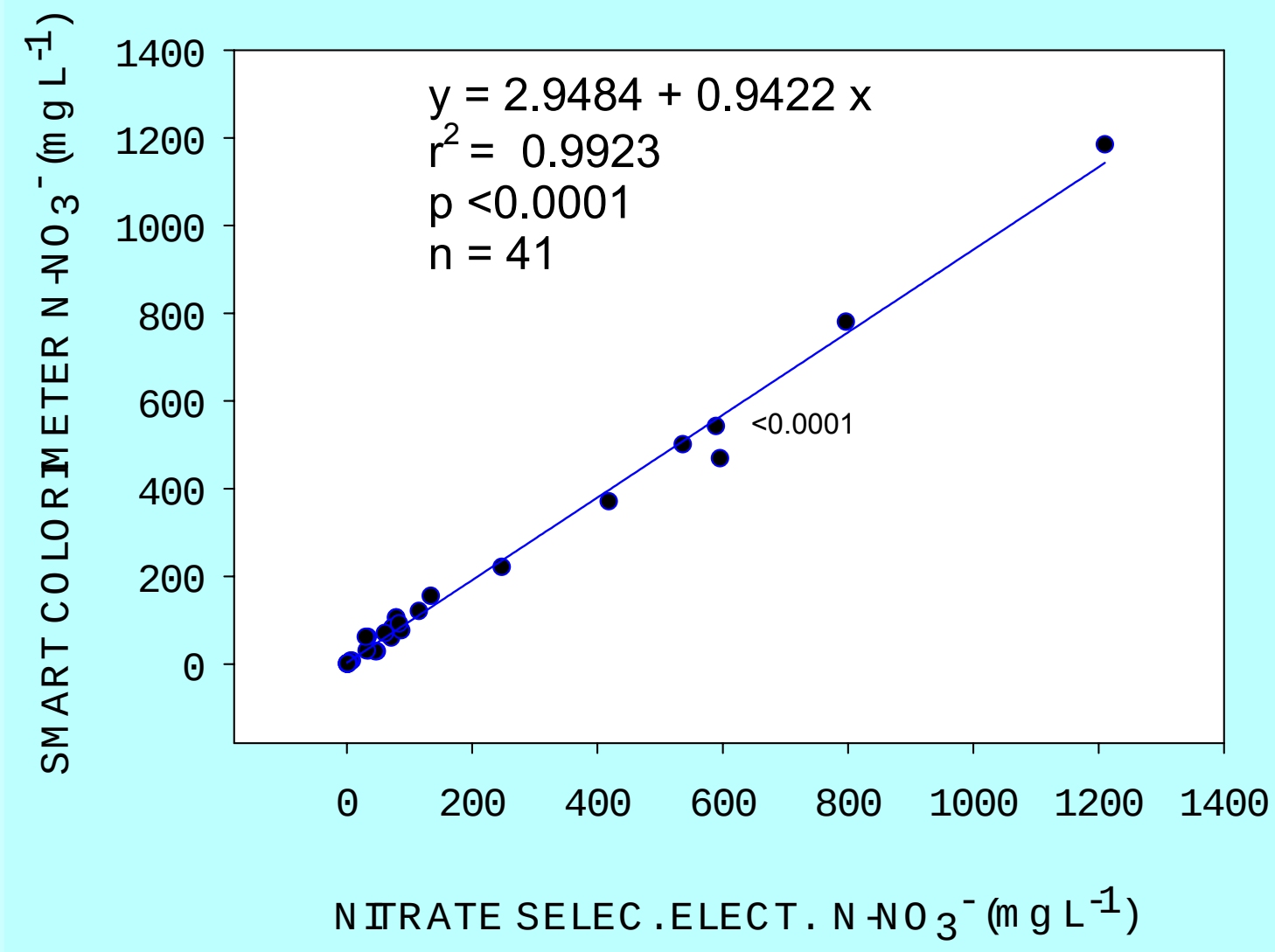
OVERALL COEFICIENT OF VARIATION FOR FRESH AND DRY TISSUE

	STRIP	CARDY	SMART	NSE	SALICYLIC
DRY	12.6	10.6	6.8	2.2	18.3
FRESH	26.8	16.0	31.9	15.5	38.3

DILUTION RATE TEST FOR NSE METHOD WITH DRY LETTUCE TISSUE



CORRELATION BETWEEN SMART COLORIMETER AND NITRATE SELECTIVE ELECTRODE N-NO3- READINGS IN NUTRIENT SOLUTION



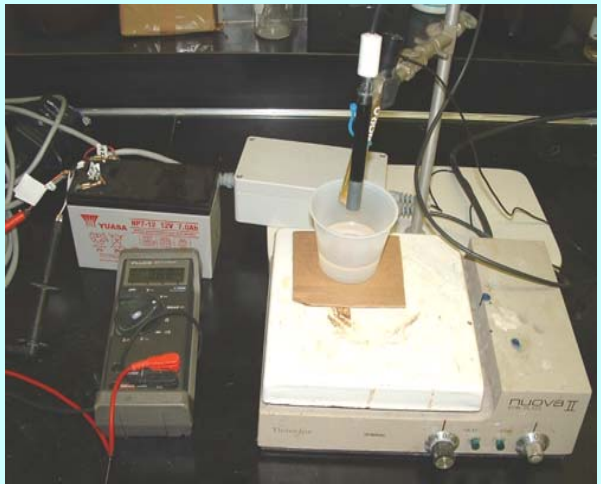
CONCLUSIONS

NO3 STRIP TEST

CARDY ION METER

SMART COLOR.

NITRATE ESPECIFIC ELECTRODE



SALICYLIC ACID

CONCLUSIONS

METHOD	ADVANTAGE	DISADVANTAGE
NO3 STRIP TEST	1- QUICK; 2- CHEAP IN A SHORT TERM; 3- DIRECT RESULT; 4- GOOD FOR A FEW SAMPLES, SPECIALLY FOR NUTRIENT SOLUTION.	1- EXPENSIVE IN THE LONG TERM; 2-IT NEDDS EXACT DILUTION TO FIT IN THE READING RANGE; 3- HIGH VARIABILITY / LOW ACCURACY.

CONCLUSIONS

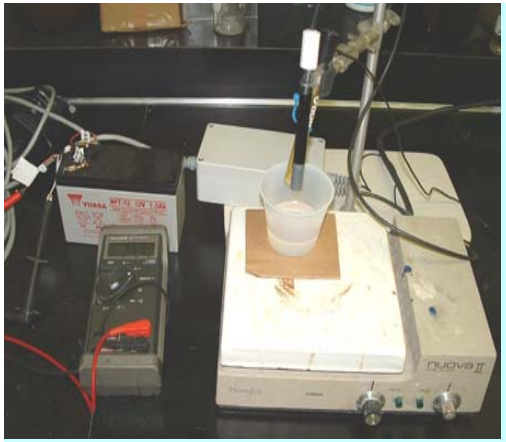
METHOD	ADVANTAGE	DISADVANTAGE
CARDY ION METER	1- FAST; 2- DIRECT READING; 3- HUGE RANGE OF READING; 4- CHEAP; 5- GOOD FOR FIELD TESTS.	1- ERRACTIC READINGS ON LOW NITRATE CONCENTRATION; 2-SUFFER HIGH INTERFERENCE OF THE SOLUTION IONIC STRENGTH; 3- LOW REPRODUCTIVENESS OF THE CALIBRATION.

CONCLUSIONS

METHOD	ADVANTAGE	DISADVANTAGE
SMART COLORIMETER	1- FAST FOR A FEW SAMPLES; 2- HAS GOOD ACCURACY FOR LOW NITRATE CONCENTRATIONS;	1- VERY EXPENSIVE; 2- USE POISON REAGENTS; 3- IT NEEDS EXACT DILUTION/SHORT RANGE. 4- SUFFER INTERFERENCE OF THE EXTRACT COLOR.

CONCLUSIONS

METHOD	ADVANTAGE	DISADVANTAGE
SALICYLIC ACID	1- IT USES A COMMOM PHOTOCOLORIMETER; 2- CHEAP; 3- HIGH RANGE OF READING; 4- GOOD FOR A LOT OF SAMPLES.	1- TIME CONSUMPTION WITH A FEW SAMPLES; 2- IT IS NOT SUITABLE FOR LOW NITRATE CONCENTRATIONS; 3- IT NEEDS CALIBRATION CURVE.



CONCLUSIONS

METHOD	ADVANTAGE	DISADVANTAGE
NSE	1- CHEAP A LONG TERM; 2- VERY ACCURATE IN A HUGE RANGE OF READING; 3- NO DILUTION; 4- FAST FOR MANY SAMPLES; 5- GOOD FOR THE MOST TYPES OF MATERIAL	1- TIME CONSUMPTION FOR A FEW SAMPLES; 2- IT NEEDS CALIBRATION CURVE;