CHN Elemental Analyzer Labs: Successes (few), Failures (many), and Future Activities

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Arizona Western College

Yuma, AZ


Las Vegas
Glen Canyon Dam
San Juan River
Grand Canyon
Little Colorado River
Flagstaff
Colorado River
Phoenix
ARIZONA
NEW MEXICO
AWC
MEXICO
Tucson

Arizona Western College

- Public Two-Year College
- 6,000 FTSE (Full-time Student Equivalents)
- Partnerships with NAU & UA
- New 40,000ft^2 Agriculture-Science bldg., Spring 2007

Ocotillo

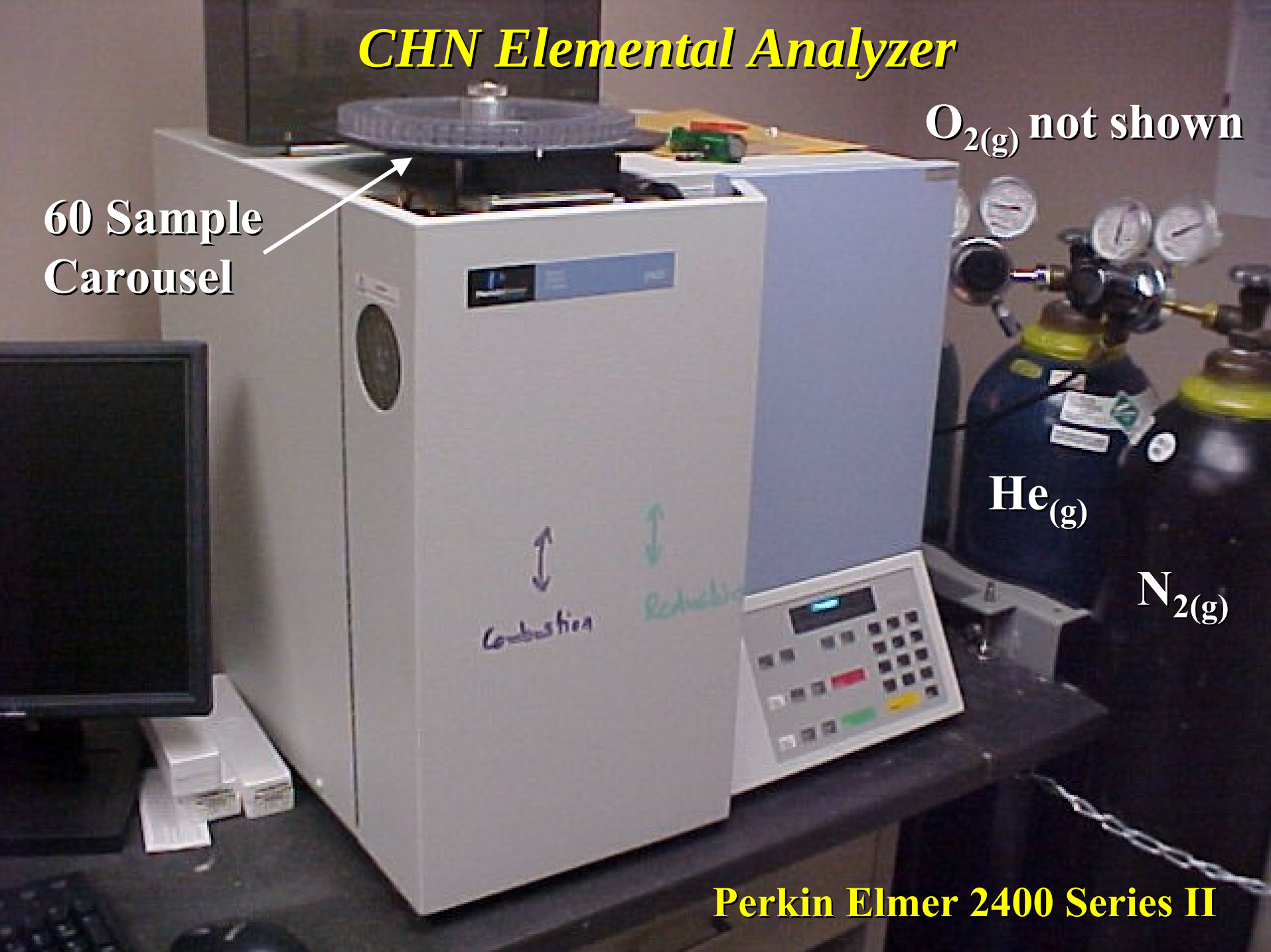
Presentation Topics

1) Overview of CHN analyzer

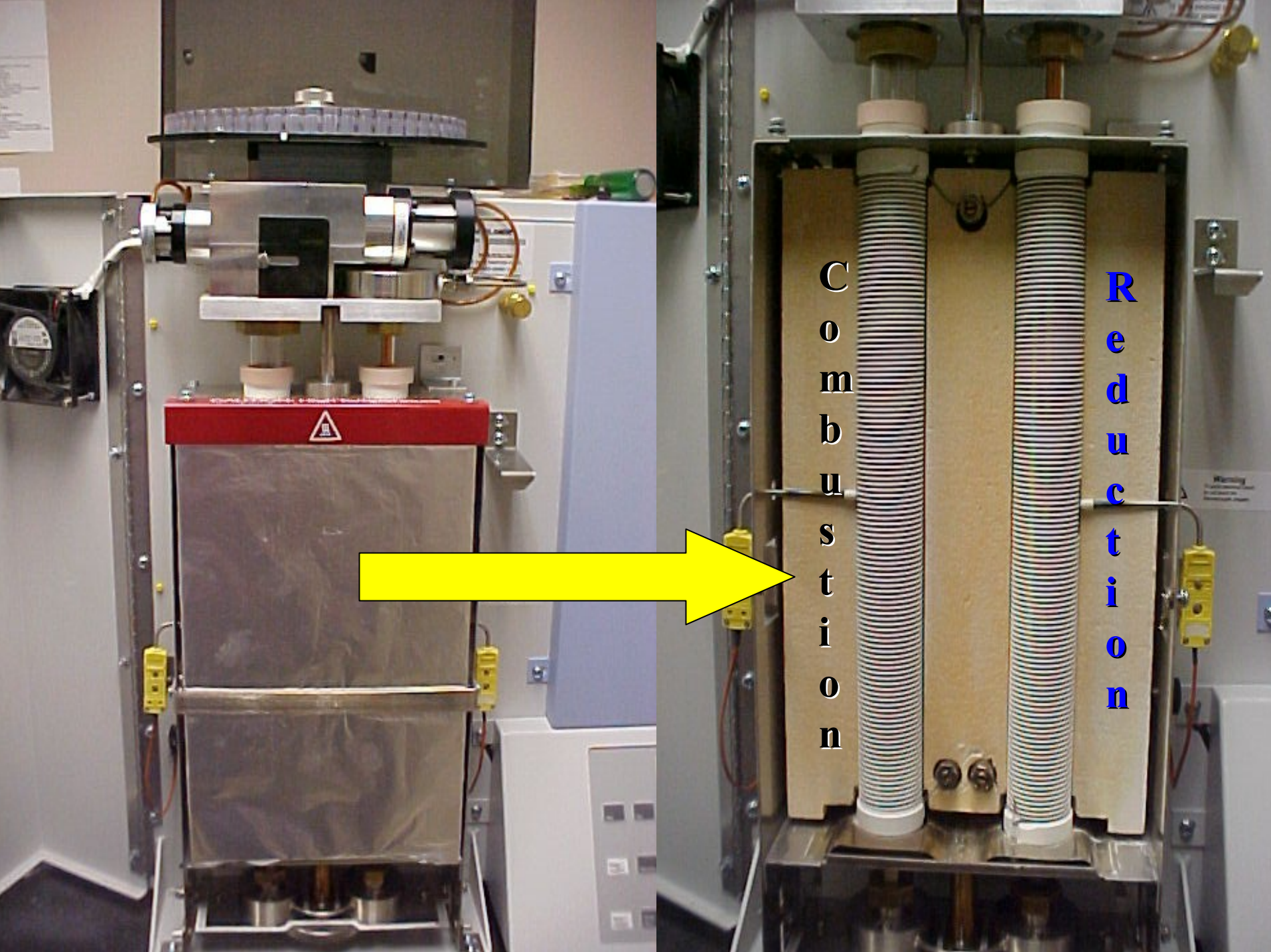
2) CHN Labs in

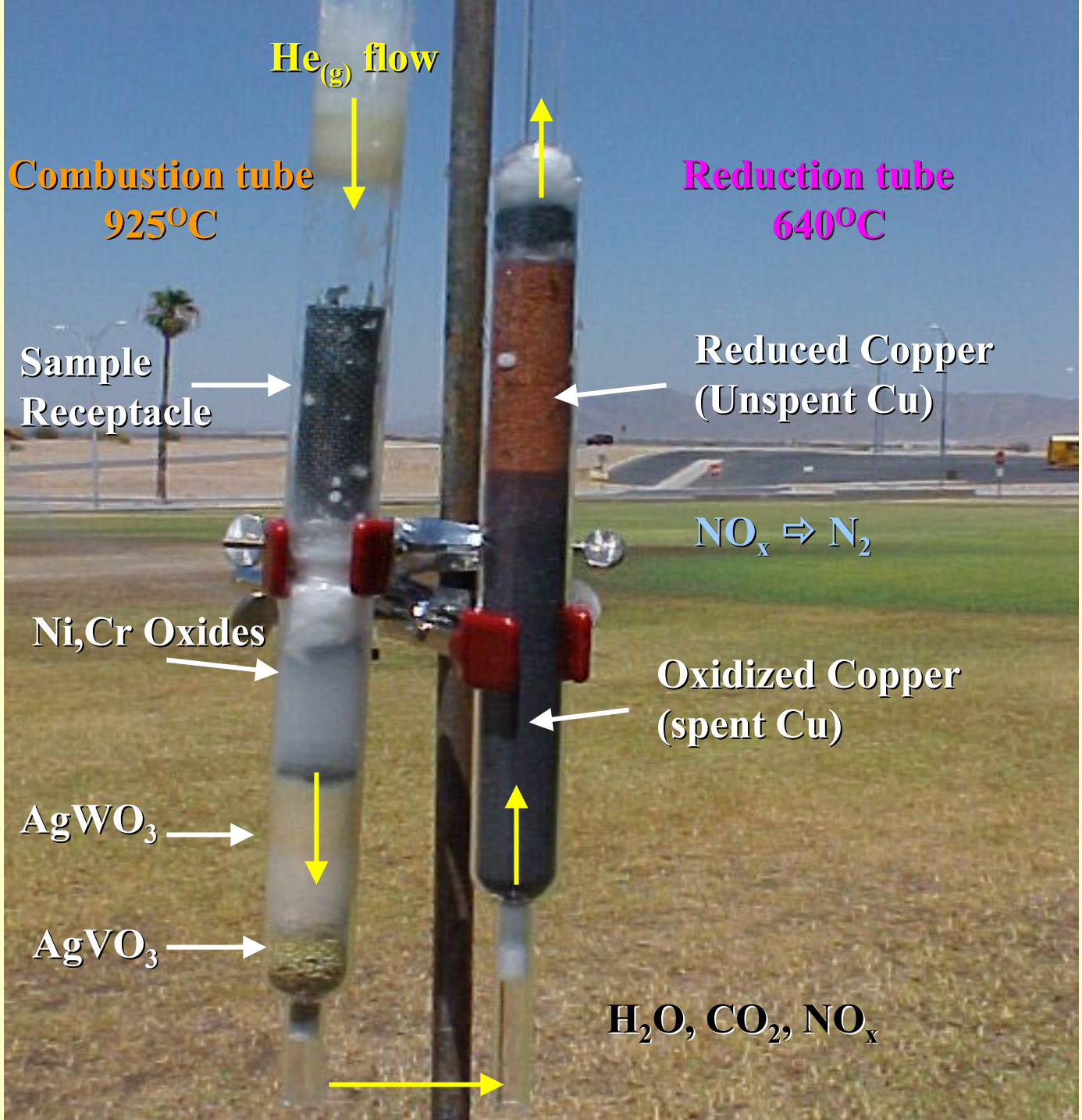
- **✦Organic Chemistry**
  - **-assessment questions**
- **✦Environmental Science**

3) Future Activities


CHN Elemental Analyzer
$O_{2(g)}$ not shown
60 Sample Carousel
$He_{(g)}$
$N_{2(g)}$
Perkin Elmer 2400 Series II

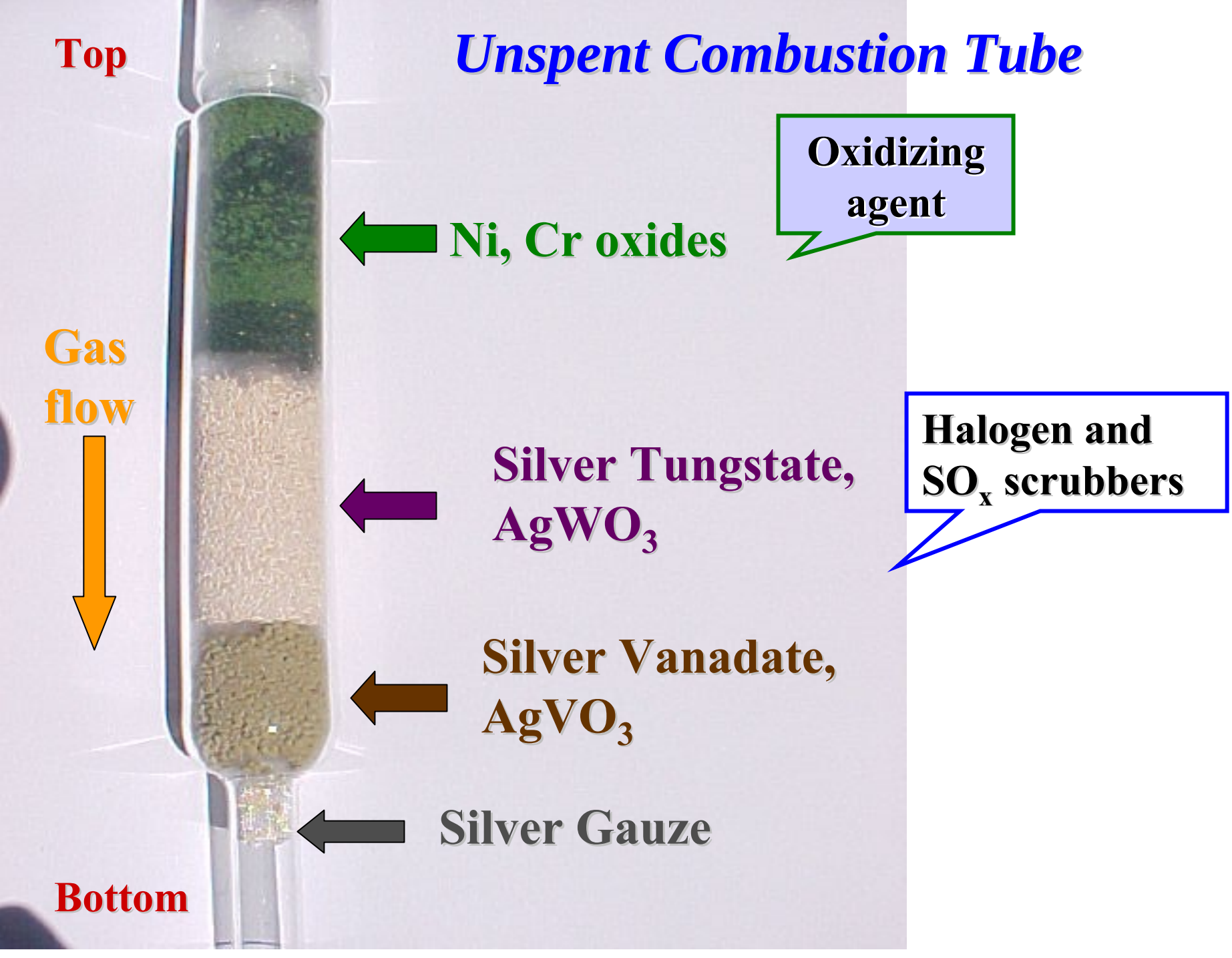

Mass (mg)
% C
% H
% N
2mg < sample mass < 5mg
2.95
1.5
0.21
Tin cup
4.75
1.2
0.14
1.4
0.18

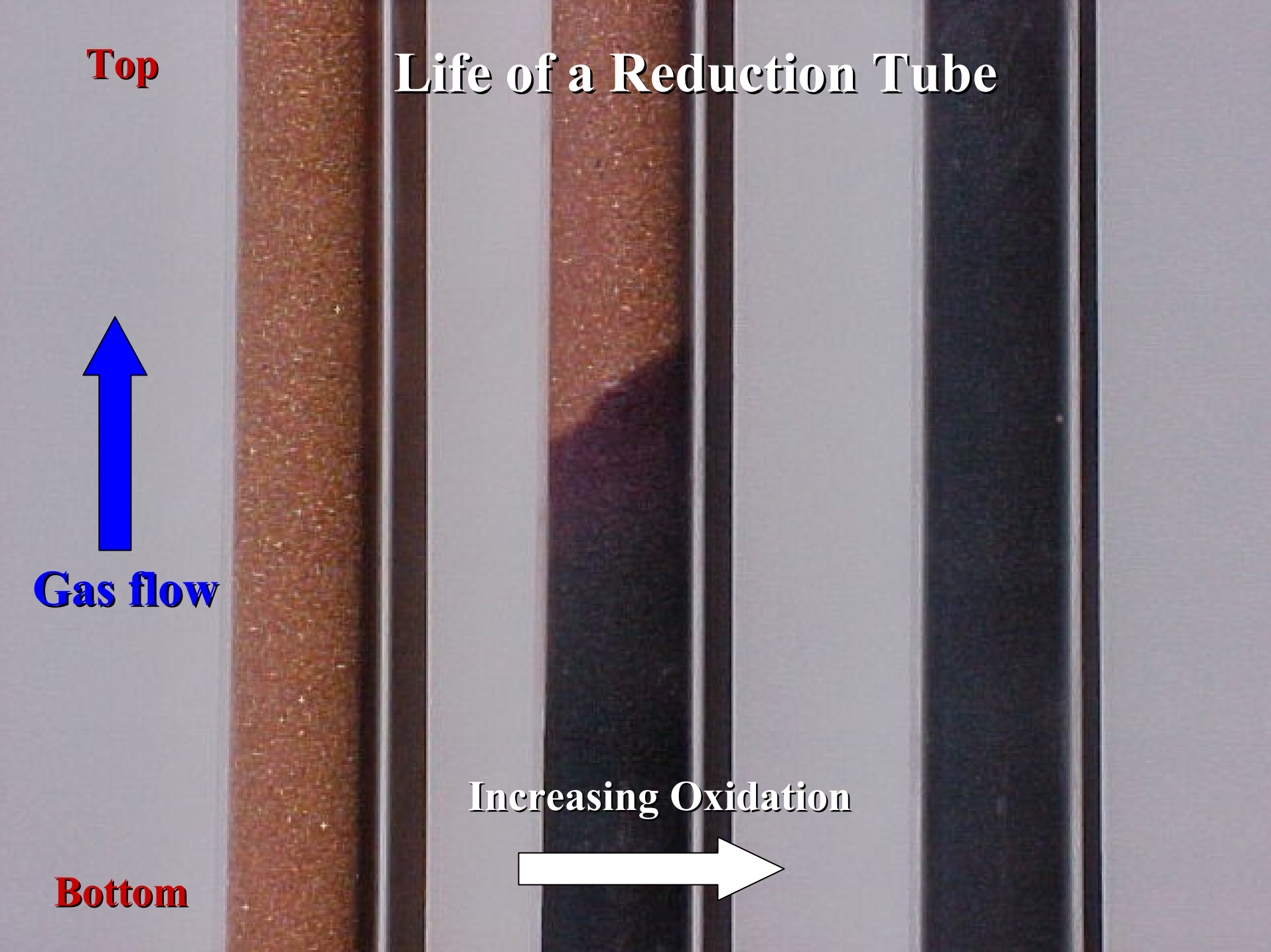

Combustion
Reduction

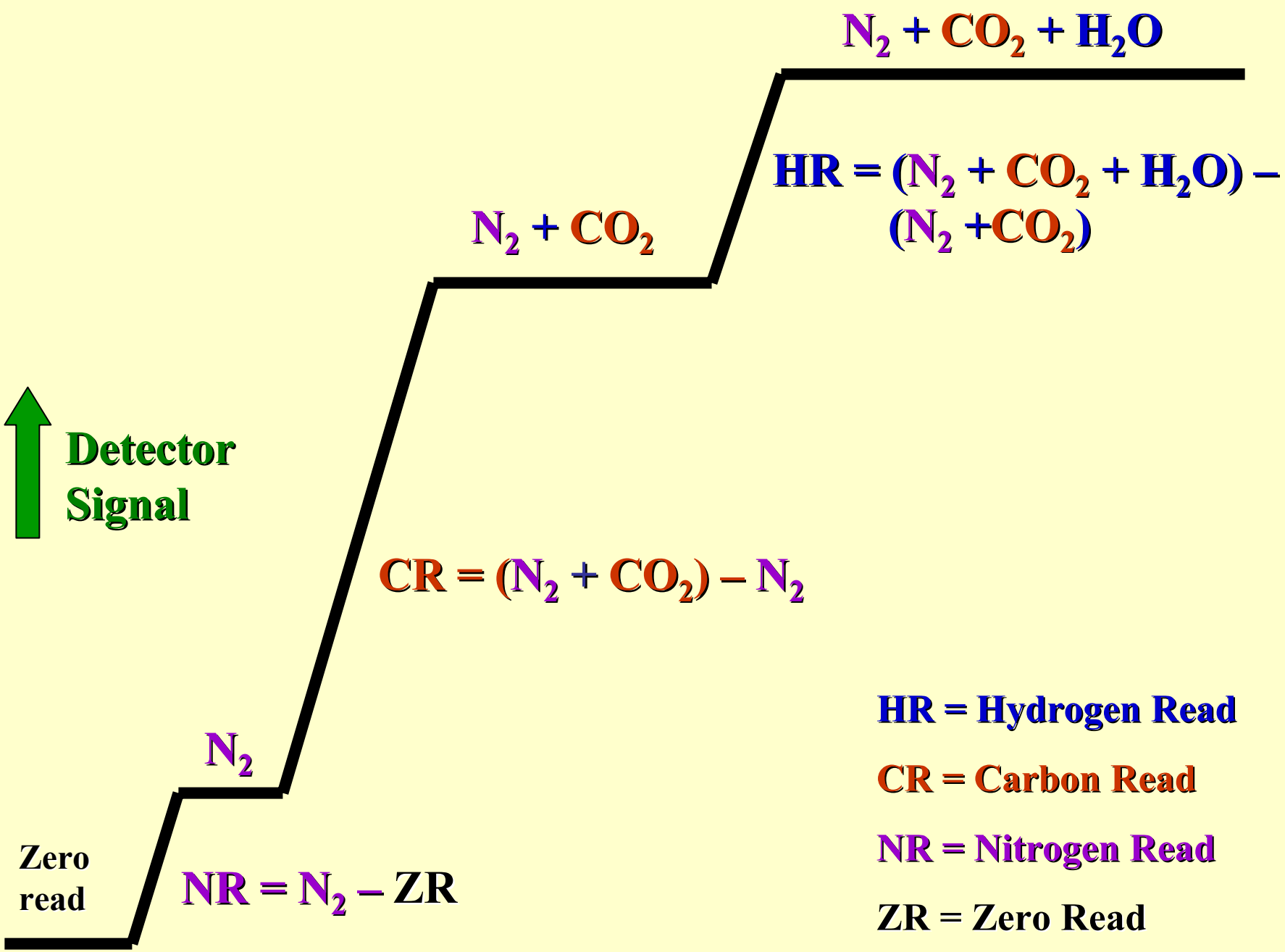


oxidizing agent

halogen, SO_x scrubbers


Top
Unspent Combustion Tube
Oxidizing agent
Ni, Cr oxides
Gas flow
Silver Tungstate, $AgWO_3$
Halogen and SO_x scrubbers
Silver Vanadate, $AgVO_3$
Silver Gauze
Bottom


Top
Life of a Reduction Tube
Gas flow
Increasing Oxidation
Bottom


$N_2 + CO_2 + H_2O$
$HR = (N_2 + CO_2 + H_2O) - (N_2 + CO_2)$
$N_2 + CO_2$
Detector Signal
$CR = (N_2 + CO_2) - N_2$
N_2
Zero read
$NR = N_2 - ZR$
HR = Hydrogen Read
CR = Carbon Read
NR = Nitrogen Read
ZR = Zero Read

CHN Postlab Assessment Question

A. In lab last week you reacted *tert*-butyl chloride with phenol to form in different amounts the ortho and para alkylated products. The petro ether solvent was cooled, a precipitate formed, and was dried. Mass %C and %H data from a CHN analysis agree with the Merck Index. You conclude that the precipitate is ρ-*tert*-butyl phenol. Is this a good idea to make such a conclusion without having also done a melting point? *Explain your answer.*

A weak majority of students concluded correctly that a CHN analysis, in combination with melting range determination, are needed since the ortho and para monosubstituted products are isomers.

Incorrect answers included failure to calculate mass % data correctly and the unquestionable belief that an instrument cannot be wrong.

CHN Postlab Assessment Question

A. Without doing a melting point could the CHN analysis allow you to rule out the formation of an appreciable amount of 2,4-di-*tert*-butyl phenol? *Explain*.

All students answered that CHN mass % data in theory could rule out the formation, in appreciable amounts, of 2,4-di-*tert*-butyl phenol (%C: 80% vs. 81.6%; %H: 9.3% vs. 10.7%).

CHN Postlab Assessment

Halogens on the benzene ring act as o, ρ directors. So nitration of bromobenzene could form the following three products: *1-bromo-2-nitrobenzene* (mp: 40-42^{O}C), *1-bromo-4-nitrobenzene* (mp: 124-126^{O}C), and *1-bromo-2,4-dinitrobenzene* (mp: 71-73^{O}C).

<u>A.</u> CHN analysis of 2.65mg recrystallized product gave 37.7%C, 2.04%H, and 5.71%N. The recrystallized product's melting point is 121-123^{O}C. Which of the three nitro products was formed in greatest amounts?

Correct = 80% **Incorrect = 20%**

CHN Postlab Assessment

B. Suppose the melting point of 1-bromo-2,4-dinitro-benzene was unknown. It could very well be 124-126^{O}C. And maybe not. Hmmm…how can CHN analysis rule out 1-bromo-2,4-dinitrobenzene as the product formed in greatest amount? *Explain.*

All students answered in one way or another that CHN analysis could rule out the formation of 1-bromo-2,4-dinitrobenzene based on a greater mass % N relative to the other two possible products, 11.3% vs. 6.9% respectively.

Future Postlab Assessment Question: Negative Nitrogen Mass Percent

A. As you know the CHN analyzer has given negative N mass percents lately. The nitrogen weight percent (%N) is calculated according to the equation below.

$$\%N = \frac{[(NR - ZR) - NB] \times 10^2}{(SW \times N\ KF)}$$

where NB = nitrogen blank, SW = sample weight (in mg), and N KF (nitrogen K factor) = 5.751. From the equation above determine how the %N calculation is coming out negative.

B. Give a brief description how you could determine if a high nitrogen blank (NB) reading is a result of nitrogen contamination in the oxygen tank.

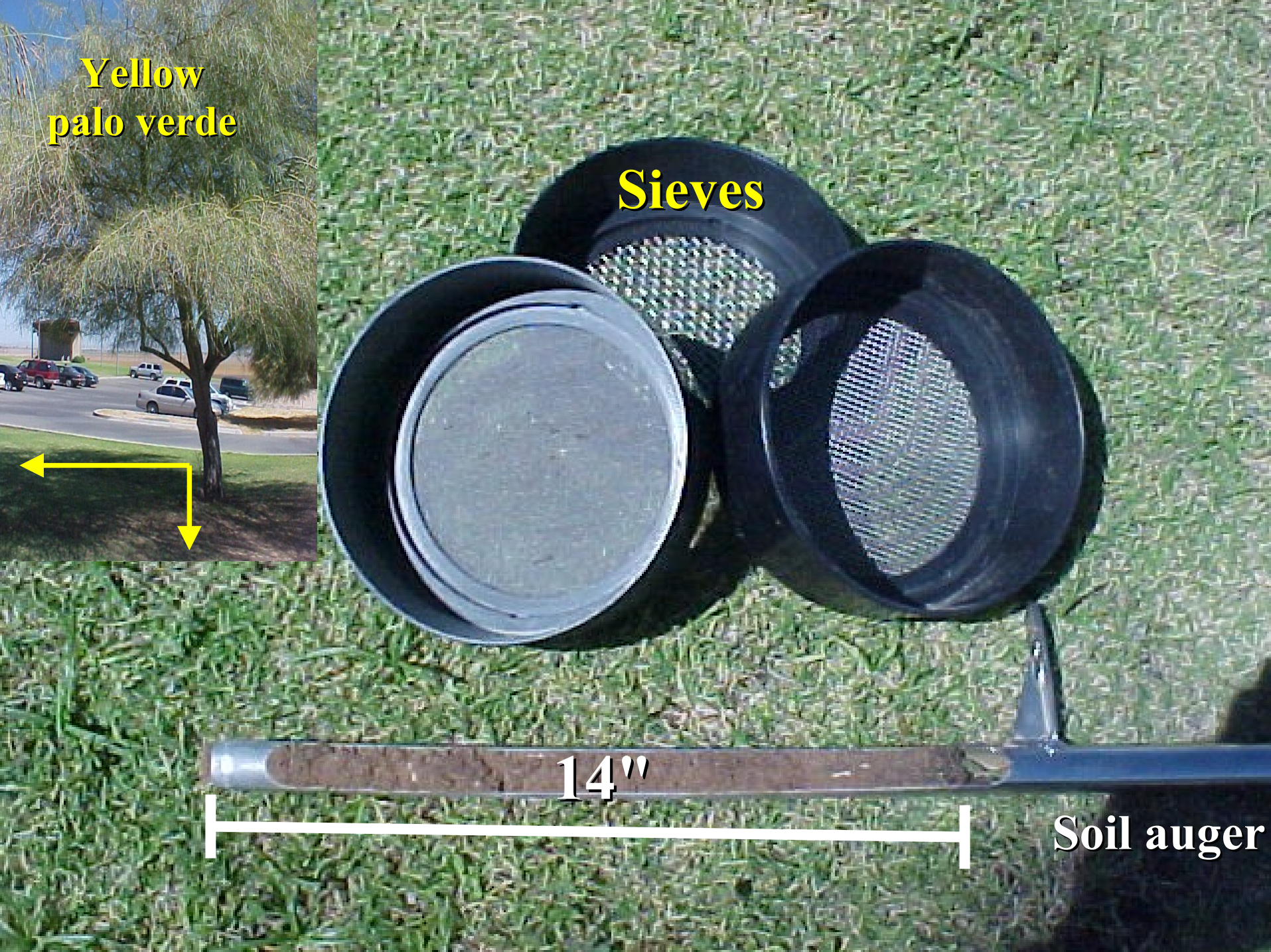
Another Future Postlab Assessment Question: Reduction Tube

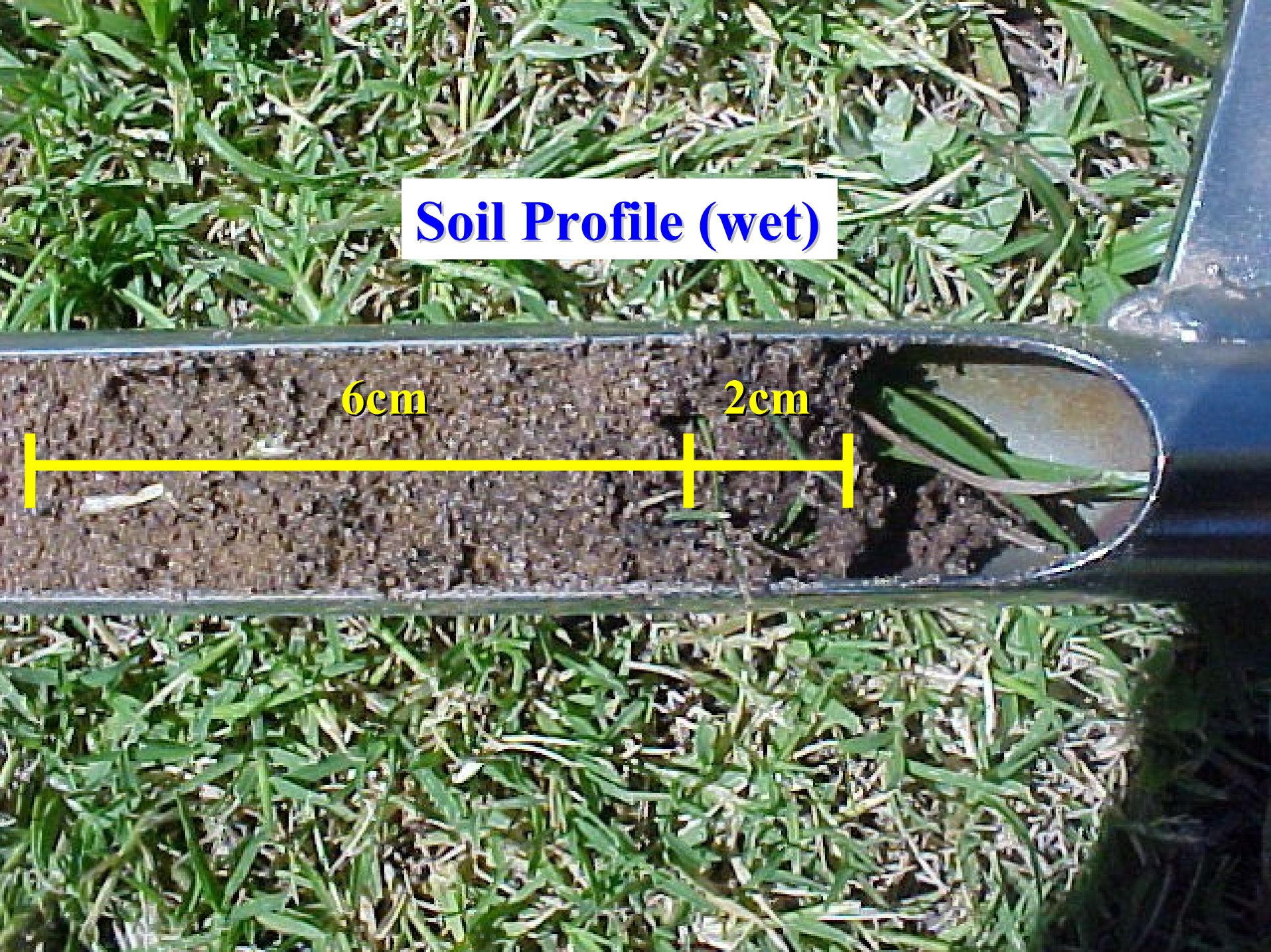
The reduction tube used in CHN analysis is filled with zero valent (elemental) copper. Ignoring cost of materials, propose a reason why copper is used instead of either of copper's period 4 transition metal neighbors- nickel or zinc.


Nitrogenous soot deposits in tailpipe?
CHN Conclusion: No

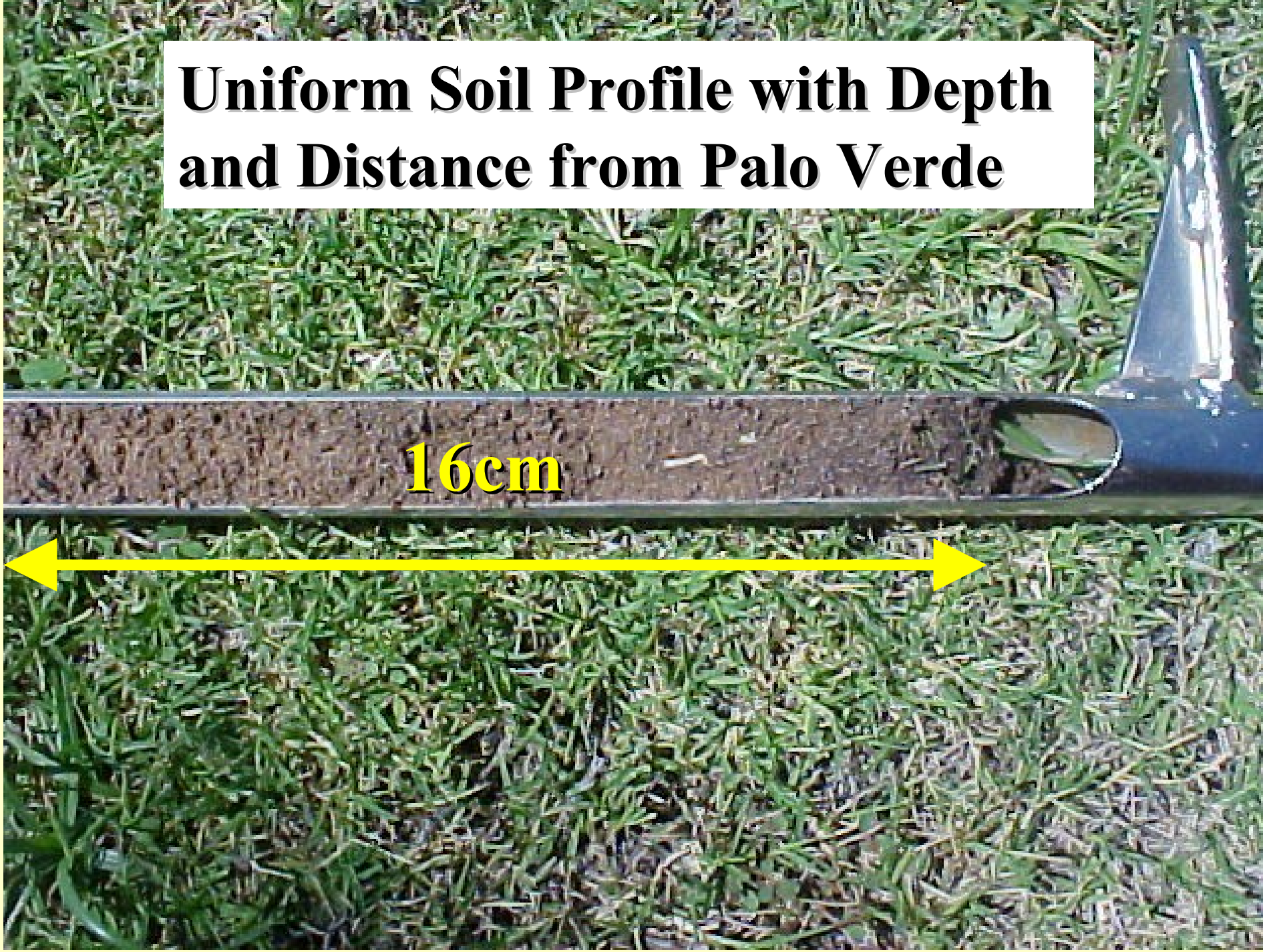
CHN Labs in Environmental Science

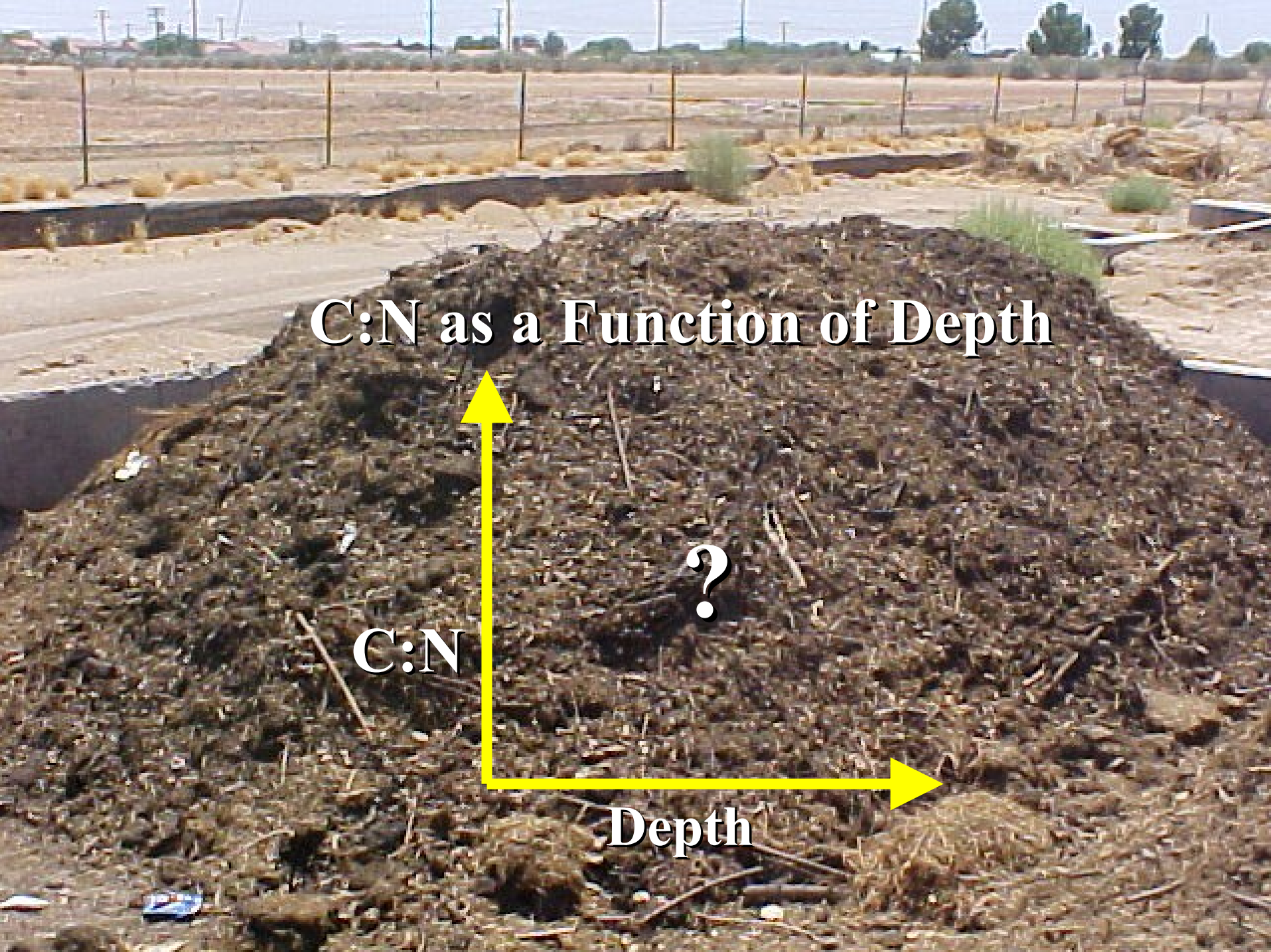
- **C:N profile in soil surrounding palo verde, a leguminous tree native to the Sonoran desert**
- **C:N profiles in an aged compost pile**


Yellow
palo verde
Sieves
14"
Soil auger


Soil Profile (wet)
6cm
2cm

Uniform Soil Profile with Depth and Distance from Palo Verde




C:N as a Function of Depth
?
C:N
Depth

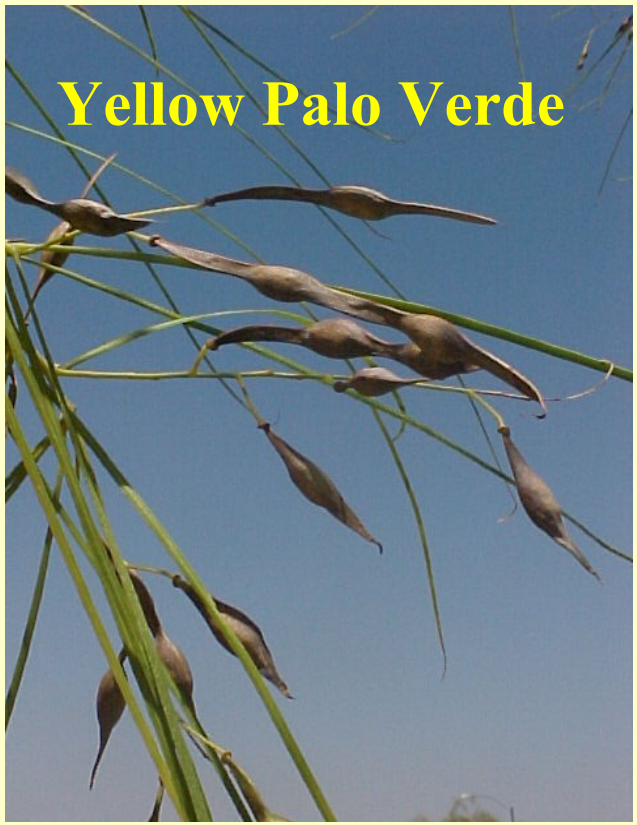
CHN Results for Edamame

Mass % (average, 3 samples)

C: 56.1 H: 8.6 N: 6.8

Photo source: www.hort.purdue.edu/newcrop/ncnu02/v5-432.html

Statistical Difference in C:N?



Blue Palo Verde

%C = 52.1

%N = 5.6

C:N = 9.3

%C = 51.6

%N = 2.2

C:N = 24

Acknowledgements

1) National Science Foundation (NSF)

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2) All my students….they do the hard work & receive lil' credit