Food and Agriculture Organization of the United Nations

LATSOLAN
LATIN AMERICAN SOIL LABORATORY NETWORK

Pathways to precision in soil analysis: advancing soil laboratories in Latin America and the Caribbean

Caminos hacia la Precisión en el Análisis de Suelos: avance de los Laboratorios de Suelos en América Latina y el Caribe

Calibration

Daniel Vidal Pérez

WORKSHOP
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GLOBAL SOIL PARTNERSHIP

- Lab Glassware (volumetric);
- Equipments (Weigh balance, UV/Vis, Flame Photometer, etc);
- Signal x Results – Calibration curve

LAB GLASSWARE (VOLUMETRIC)

- Some examples are pipets, volumetric flasks and burets;
- The value marked on a piece of glassware is called the nominal value;
- The true value is usually slightly different from the nominal value;
- The key measuring device in the laboratory is the analytical balance.

LAB GLASSWARE (VOLUMETRIC)

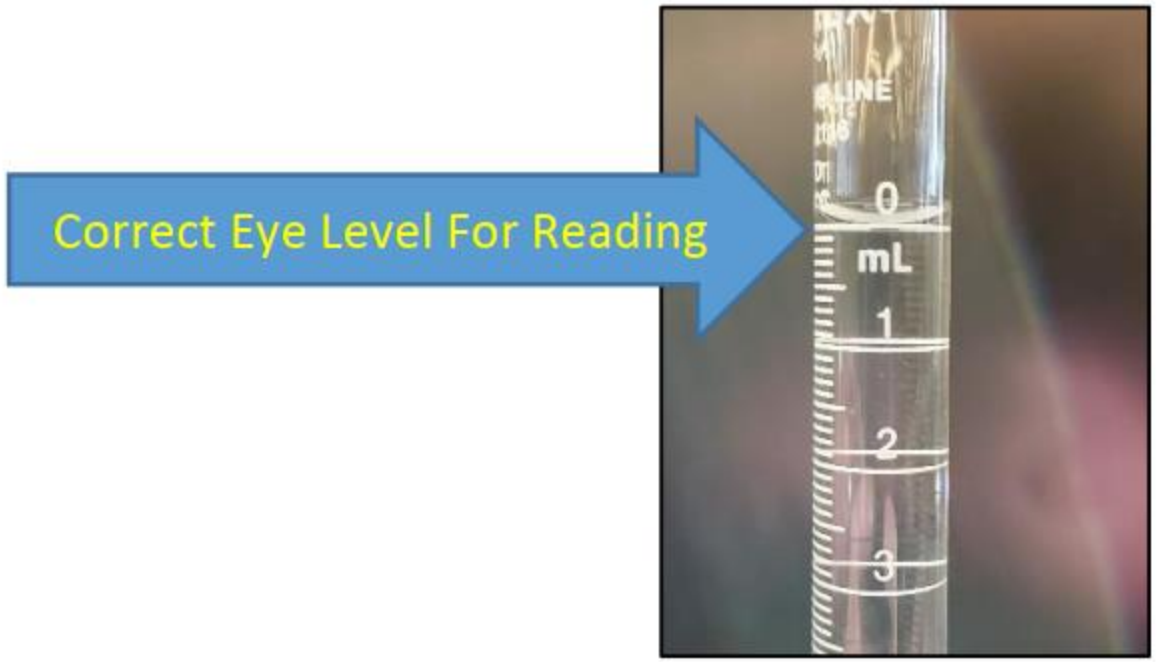
- To Contain vs. To Deliver;

A. The volume of a pipet is determined by weighing the water **delivered** into a clean dry container.
From the weight and the density of water one can calculate the true volume delivered;

B. To calibrate a volumetric flask, it is first weighed empty, clean and dry.
It is then **filled** to the mark with water and again weighed.
As before, the volume is calculated from the weight of water and its density

LAB GLASSWARE (VOLUMETRIC)

- An example: Calibration of a Burette

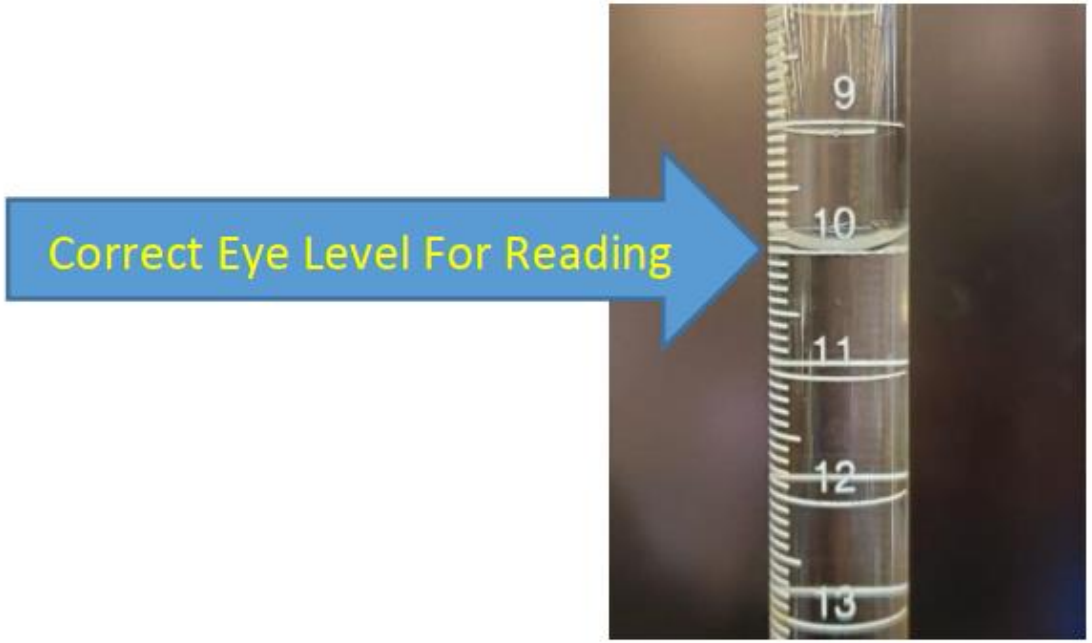


Using the burette, deliver 10.00 mL of the distilled water into the pre-weighed 100-mL volumetric flask.

LAB GLASSWARE (VOLUMETRIC)

- An example: Calibration of a 50 mL Burette

Figure 5. Final Volume: 10.00-mL Reading



Record the mass of the filled volumetric flask.

LAB GLASSWARE (VOLUMETRIC)

- An example: Calibration of a Burette

Density of Water at 25.0 ºC (from Table 5): 0.997044 g/mL

Mass of Water and Volumetric Flask (g): **78.4294** g

Mass of Empty 100-mL Volumetric Flask (g): − **68.3996** g

Mass of Water (g): = **10.0298** g

$$\textbf{Density} = \frac{\text{Mass}}{\text{Volume}} \qquad \textbf{Volume} = \frac{\text{Mass}}{\text{Density}}$$

$$\textbf{Volume} = \frac{10.0298\text{g}}{0.997044\text{g/mL}} = 10.0595\ \text{mL}$$

Table 5. Density of Water at Various Temperatures

Temperature (ºC)	Density (g/mL)	Temperature (ºC)	Density (g/mL)	Temperature (ºC)	Density (g/mL)
19.0	0.998405	21.6	0.997860	24.2	0.997246
19.2	0.998365	21.8	0.997815	24.4	0.997196
19.4	0.998325	22.0	0.997770	24.6	0.997146
19.6	0.998285	22.2	0.997724	24.8	0.997095
19.8	0.998265	22.4	0.997678	25.0	0.997044
20.0	0.998203	22.6	0.997632	25.2	0.996992
20.2	0.998162	22.8	0.997585	25.4	0.996941
20.4	0.998120	23.0	0.997538	25.6	0.996888
20.6	0.998078	23.2	0.997490	25.8	0.996836
20.8	0.998035	23.4	0.997442	26.0	0.996783
21.0	0.997922	23.6	0.997394	26.2	0.996729
21.2	0.997948	23.8	0.997345	26.4	0.996703
21.4	0.997904	24.0	0.997296		

EQUIPMENTS (WEIGH BALANCE)

- Factors that affect the performance of analytical balances:

1. acceleration of gravity;
2. ambient temperature;
3. humidity;
4. barometric pressure;
5. altitude, etc

- Conclusion: Calibration can only be given at the place of installation

EQUIPMENTS (WEIGH ANALYTICAL BALANCE)

- External calibration is a manual process that requires the user to place an approved set of weighing scale weights on the balance and set their weight as the standard;

- Performance Check: 1 mg, 2 mg, 5 mg, 10 mg and 20 mg calibration standard weights should be placed separately on the top pan balance after the auto calibration process, and the measurements noted in the performance check log.

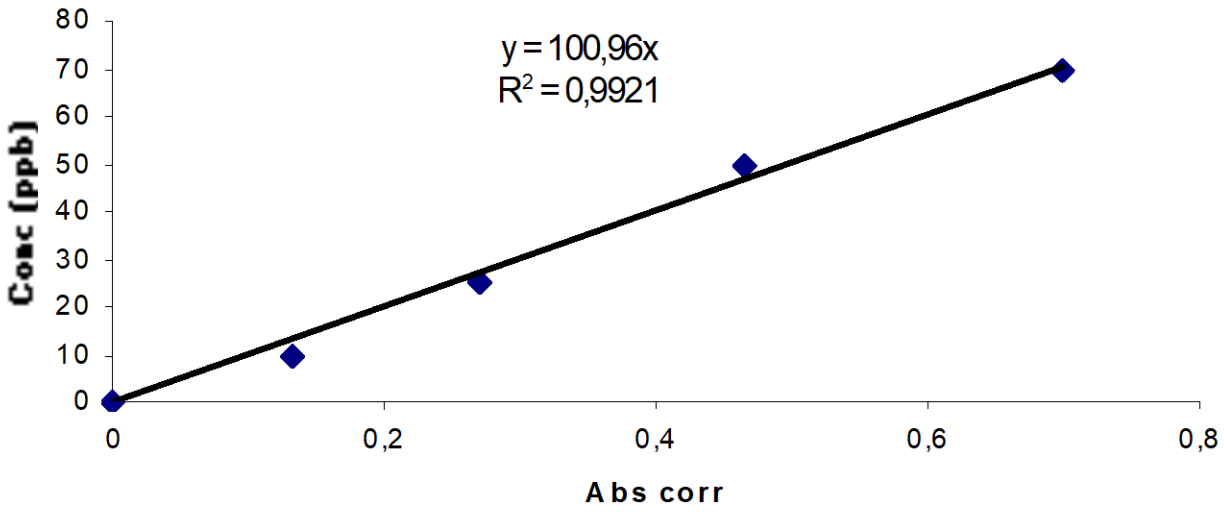
- To meet the traceable standard, these measurements need to remain within the 0.1% of the actual mass value of each weight.


40.3305

Signal x Results – CALIBRATION CURVES

- UV-Vis spectrophotometer example (Absorbance reading)

Abs	Abs Corr	Conc
0,03591	0	0
0,168	0,13209	10
0,3068	0,27089	25
0,5009	0,46499	50
0,7341	0,69819	70



Signal x Results – CALIBRATION CURVES

- Calibration curve MUST include ZERO?

RESUMO DOS RESULTADOS

Estatistica de regressão	
R múltiplo	0,996815
R-Quadrad	0,99364
R-quadrad(	0,991519
Erro padrã	2,653078
Observaçõ	5

ANOVA

	gl	*SQ*	*MQ*	*F*	*F de significação*
Regressão	1	3298,883532	3298,884	468,6698	0,000215697
Resíduo	3	21,11646806	7,038823		
Total	4	3320			

	Coeficientes	*Erro padrão*	*Stat t*	*valor-P*	*95% inferiores*	*95% superiores*
Interseção	-1,63276	1,918315169	-0,85114	0,457236	-7,737696562	4,472184937
Abs Corr	104,1808	4,812316125	21,64878	0,000216	88,86583349	119,4957376

Conc = 104.18 Abs – 1.63

Can I force to pass 0.00?

Signal x Results – CALIBRATION CURVES

- Calibration curve could include ZERO

RESUMO DOS RESULTADOS						
Estatística de regressão						
R múltiplo	0,996044					
R-Quadrad	0,992104					
R-quadrad(	0,742104					
Erro padrã	2,560062					
Observaçõ	5					
ANOVA						
	gl	*SQ*	*MQ*	*F*	*F de significação*	
Regressão	1	3293,784326	3293,784	502,5672	0,000194347	
Resíduo	4	26,21567433	6,553919			
Total	5	3320				
	Coeficiente	*Erro padrão*	*Stat t*	*valor-P*	*95% inferiores*	*95% superiores*
Interseção	0	#N/D	#N/D	#N/D	#N/D	#N/D
Abs Corr	100,9623	2,872100989	35,15276	3,91E-06	92,9880218	108,9365163

Figures and Illustrations References

- https://web.pdx.edu/~atkinsdb/teach/321/CALVOL.pdf
- https://www.kbcc.cuny.edu/academicdepartments/physci/documents/chemistry/Chem11_Lab2_CalibrationGlassware_V4_03-04-2022.pdf
- https://www.depts.ttu.edu/meatscience/docs/WhyCalibrateAnalyticalBalancesandScales-6-30-20.pdf