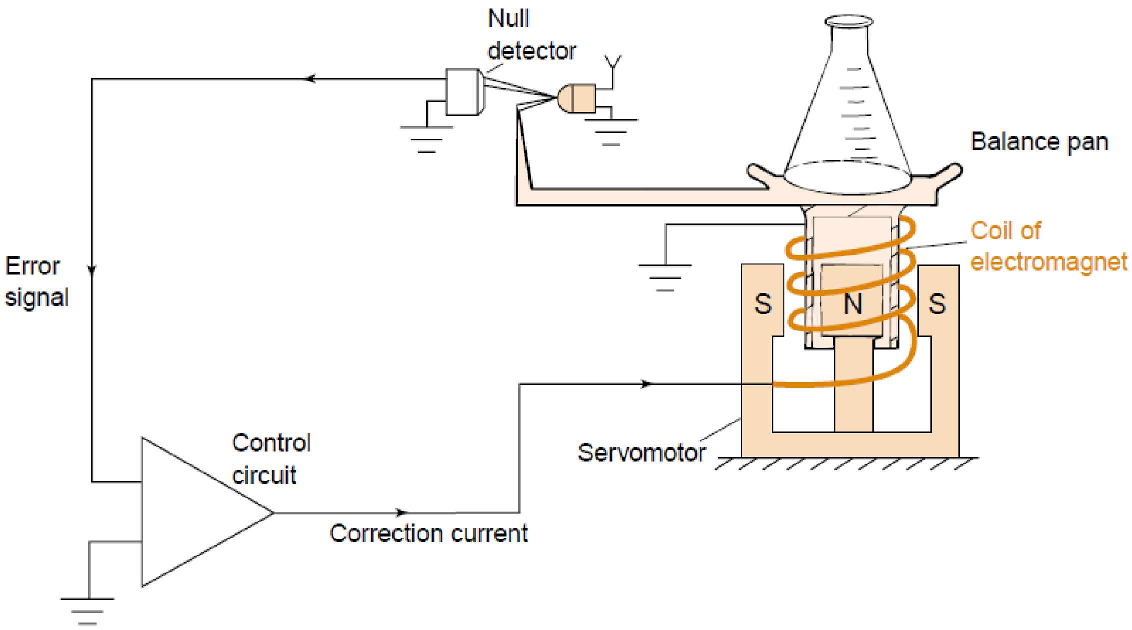
Volumetriás eszközök kalibrálása. Alapfogalmak

Elektronikus analitikai mérleg



A look at the electronic analytical balance Randall M. Schoonover Analytical Chemistry ***1982*** 54 *(8), 973A-980A*

Denomination	Tolerance (mg)		Denomination	Tolerance (mg)	
Grams	Class 1	Class 2	Milligrams	Class 1	Class 2
500	1.2	2.5	500	0.010	0.025
200	0.50	1.0	200	0.010	0.025
100	0.25	0.50	100	0.010	0.025
50	0.12	0.25	50	0.010	0.014
20	0.074	0.10	20	0.010	0.014
10	0.050	0.074	10	0.010	0.014
5	0.034	0.054	5	0.010	0.014
2	0.034	0.054	2	0.010	0.014
1	0.034	0.054	1	0.010	0.014

a. Tolerances are defined in ASTM (American Society for Testing and Materials) Standard E 617. Classes 1 and 2 are the most accurate. Larger tolerances exist for Classes 3–6, which are not given in this table.

Szárító szerek hatékonysága

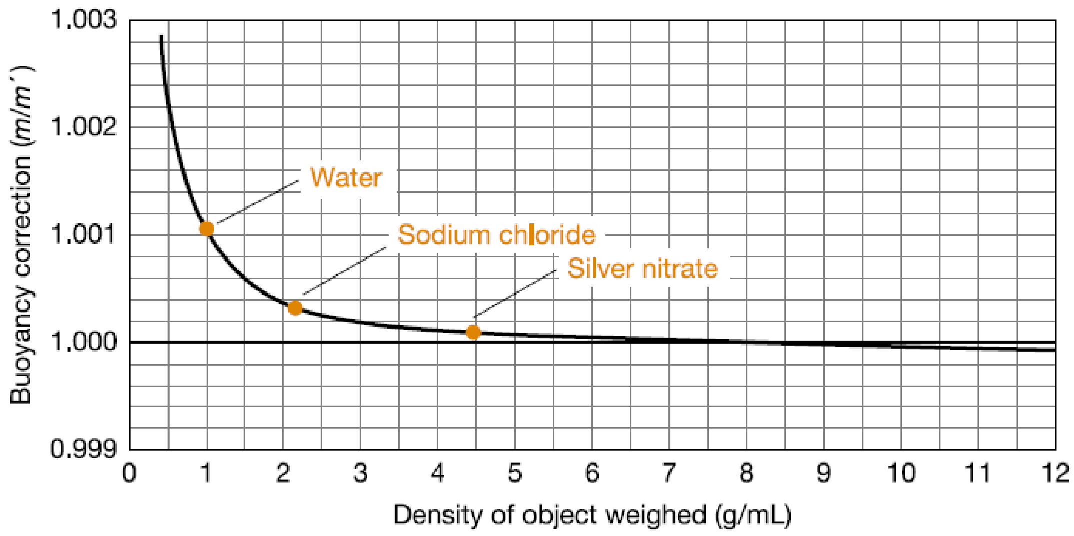
	vízmaradvány a légtérben (μg H_2O/L)
$Mg(ClO_4)_2$	0.2
$Mg(ClO_4)_2 \cdot 1\text{–}1.5H_2O$	1.5
BaO	2.8
Al_2O_3	2.9
P_4O_{10}	3.6
$CaSO_4$	67
SiO_2	70

Efficiency and Temperature Dependence of Water Removal by Membrane Dryers Kristen J. Leckrone and, John M. Hayes Analytical Chemistry ***1997*** 69 *(5), 911-918*

Felhajtó erő

$$m = \frac{m'\left(1 - \frac{d_a}{d_w}\right)}{\left(1 - \frac{d_a}{d}\right)}$$

d_a – levegő sűrűsége (0,0012 g/mL , 1 bar, 25 °C)
d_w – a kalibrációs súlyok sűrűsége (8,0 g/mL)
d- a mért anyag sűrűsége (g/mL)
m'- a mért anyag tömege (g)
m- a mért anyag valós tömege a vákuumba (g)



A pure compound called "tris" is used as a *primary standard* to measure concentrations of acids. The volume of acid required to react with a known mass of tris tells us the concentration of the acid. Find the true mass of tris (density = 1.33 g/mL) if the apparent mass weighed in air is 100.00 g.

SOLUTION Assuming that the balance weights have a density of 8.0 g/mL and the density of air is 0.001 2 g/mL, we find the true mass by using Equation 2-1:

$$m = \frac{100.00\ \text{g}\left(1 - \dfrac{0.001\ 2\ \text{g/mL}}{8.0\ \text{g/mL}}\right)}{1 - \dfrac{0.001\ 2\ \text{g/mL}}{1.33\ \text{g/mL}}} = 100.08\ \text{g}$$

Unless we correct for buoyancy, we would think that the mass of tris is 0.08% less than the actual mass and we would think that the molarity of acid reacting with the tris is 0.08% less than the actual molarity.

Volumetriás üvegeszközök térfogati hibája

Buret volume (mL)	Smallest graduation (mL)	Tolerance (mL)
5	0.01	±0.01
10	0.05 *or* 0.02	±0.02
25	0.1	±0.03
50	0.1	±0.05
	.2	±0.10

Table 2-4 Tolerances of Class A transfer pipets

Volume (mL)	Tolerance (mL)
0.5	±0.006
1	±0.006
2	±0.006
3	±0.01
4	±0.01
5	±0.01
10	±0.02
15	±0.03
20	±0.03
25	±0.03
50	±0.05
100	±0.08

Table 2-3 Tolerances of Class A volumetric flasks

Flask capacity (mL)	Tolerance (mL)
1	±0.02
2	±0.02
5	±0.02
10	±0.02
25	±0.03
50	±0.05
100	±0.08
200	±0.10
250	±0.12
500	±0.20
1 000	±0.30
2 000	±0.50

Mikropipetták térfogati pontossága

Table 2-5 Manufacturer's tolerances for micropipets

Pipet volume (μL)	At 10% of pipet volume		At 100% of pipet volume	
	Accuracy (%)	Precision (%)	Accuracy (%)	Precision (%)
Adjustable pipets				
0.2–2	±8	±4	±1.2	±0.6
1–10	±2.5	±1.2	±0.8	±0.4
2.5–25	±4.5	±1.5	±0.8	±0.2
10–100	±1.8	±0.7	±0.6	±0.15
30–300	±1.2	±0.4	±0.4	±0.15
100–1 000	±1.6	±0.5	±0.3	±0.12
Fixed pipets				
10			±0.8	±0.4
25			±0.8	±0.3
100			±0.5	±0.2
500			±0.4	±0.18
1 000			±0.3	±0.12

Táblázat az üvegeszközök térfogatának kalibrálásához

a- felhajtó erő korrekcióval

b- Felhajtó erő és boroszilikát üvegedény hőtágulási együtthatójával korrigált érték (0,0010 % K^{-1})

t, °C	d, g/mL	1 g víz térfogata	
		az adott hőmérsékleten[a]	20°C[b]
10	0.999 702 6	1.001 4	1.001 5
11	0.999 608 4	1.001 5	1.001 6
12	0.999 500 4	1.001 6	1.001 7
13	0.999 380 1	1.001 7	1.001 8
14	0.999 247 4	1.001 8	1.001 9
15	0.999 102 6	1.002 0	1.002 0
16	0.998 946 0	1.002 1	1.002 1
17	0.998 777 9	1.002 3	1.002 3
18	0.998 598 6	1.002 5	1.002 5
19	0.998 408 2	1.002 7	1.002 7
20	0.998 207 1	1.002 9	1.002 9
21	0.997 995 5	1.003 1	1.003 1
22	0.997 773 5	1.003 3	1.003 3
23	0.997 541 5	1.003 5	1.003 5
24	0.997 299 5	1.003 8	1.003 8
25	0.997 047 9	1.004 0	1.004 0
26	0.996 786 7	1.004 3	1.004 2
27	0.996 516 2	1.004 6	1.004 5
28	0.996 236 5	1.004 8	1.004 7
29	0.995 947 8	1.005 1	1.005 0
30	0.995 650 2	1.005 4	1.005 3

Hőmérséklet korrekció

$$\frac{c'}{d'} = \frac{c}{d}$$

c', d'- koncentráció és sűrűség T' hőmérsékleten

c', d'- koncentráció és sűrűség egy T hőmérsékleten

d (g/mL) = $a_0 + a_1 \times T + a_2 \times T^2 + a_3 \times T^3$

T-hőmérséklet

a_0 = 0,999 89

a_1= 5,3322 × 10^{-5}

a_2= 7,589 9 × 10^{-6}

a_3= 3, 671 9 × 10^{-8}

4-40 ° C között 5 tizedes jegyre pontos

A 0.031 46 M aqueous solution was prepared in winter when the lab temperature was 17°C. What is the molarity of the solution on a warm day when the temperature is 25°C?

SOLUTION We assume that the thermal expansion of a dilute solution is equal to the thermal expansion of pure water. Then, using Equation 2-2 and densities from Table 2-7, we write

$$\frac{c' \text{ at } 25°}{0.997\,05 \text{ g/mL}} = \frac{0.031\,46 \text{ M}}{0.998\,78 \text{ g/mL}} \Rightarrow c' = 0.031\,41 \text{ M}$$

The concentration has decreased by 0.16% on the warm day.

Reference Procedure: Calibrating a 50-mL Buret

This procedure tells how to construct a graph such as Figure 3-3 to convert the measured volume delivered by a buret to the true volume delivered at 20°C.

1. Fill the buret with distilled water and force any air bubbles out the tip. See whether the buret drains without leaving drops on the walls. If drops are left, clean the buret with soap and water or soak it with cleaning solution.[11] Adjust the meniscus to be at or slightly below 0.00 mL, and touch the buret tip to a beaker to remove the suspended drop of water. Allow the buret to stand for 5 min while you weigh a 125-mL flask fitted with a rubber stopper. (Hold the flask with a tissue or paper towel, not with your hands, to avoid changing its mass with fingerprint residue.) If the level of the liquid in the buret has changed, tighten the stopcock and repeat the procedure.
2. Drain approximately 10 mL of water at a rate <20 mL/min into the weighed flask, and cap it tightly to prevent evaporation. Allow about 30 s for the film of liquid on the walls to descend before you read the buret. Estimate all readings to the nearest 0.01 mL. Weigh the flask again to determine the mass of water delivered.
3. Now drain the buret from 10 to 20 mL, and measure the mass of water delivered. Repeat the procedure for 30, 40, and 50 mL. Then do the entire procedure (10, 20, 30, 40, 50 mL) a second time.
4. Use Table 2-7 to convert the mass of water to the volume delivered. Repeat any set of duplicate buret corrections that do not agree to within 0.04 mL. Prepare a calibration graph like that in Figure 3-3, showing the correction factor at each 10-mL interval.

Example Buret Calibration

When draining the buret at 24°C, you observe the following values:

Final reading	10.01	10.08 mL
Initial reading	0.03	0.04
Difference	9.98	10.04 mL
Mass	9.984	10.056 g
Actual volume delivered	10.02	10.09 mL
Correction	+0.04	+0.05 mL
Average correction	+0.045 mL	

To calculate the actual volume delivered when 9.984 g of water is delivered at 24°C, look at the column of Table 2-7 headed "Corrected to 20°C." In the row for 24°C, you find that 1.000 0 g of water occupies 1.003 8 mL. Therefore, 9.984 g occupies (9.984 g)(1.003 8 mL/g) = 10.02 mL. The average correction for both sets of data is +0.045 mL.

To obtain the correction for a volume greater than 10 mL, add successive masses of water collected in the flask. Suppose that the following masses were measured:

Volume interval (mL)	Mass delivered (g)
0.03–10.01	9.984
10.01–19.90	9.835
19.90–30.06	10.071
Sum 30.03 mL	29.890 g

The total volume of water delivered is (29.890 g) × (1.003 8 mL/g) = 30.00 mL. Because the indicated volume is 30.03 mL, the buret correction at 30 mL is −0.03 mL.

$$\begin{array}{rl} 1.76\ (\pm 0.03) & \leftarrow e_1 \\ +\ 1.89\ (\pm 0.02) & \leftarrow e_2 \\ -\ 0.59\ (\pm 0.02) & \leftarrow e_3 \\ \hline 3.06\ (\pm e_4) & \end{array}$$

$$e_4 = \sqrt{e_1^2 + e_2^2 + e_3^2}$$

Example Uncertainty in a Buret Reading

The volume delivered by a buret is the difference between the final reading and the initial reading. If the uncertainty in each reading is ±0.02 mL, what is the uncertainty in the volume delivered?

SOLUTION Suppose that the initial reading is 0.05 (±0.02) mL and the final reading is 17.88 (±0.02) mL. The volume delivered is the difference:

$$\begin{array}{r} 17.88\ (\pm 0.02) \\ -\ 0.05\ (\pm 0.02) \\ \hline 17.83\ (\pm e) \end{array} \qquad e = \sqrt{0.02^2 + 0.02^2} = 0.02_8 \approx 0.03$$

$$\frac{1.76\ (\pm 0.03) \times 1.89\ (\pm 0.02)}{0.59\ (\pm 0.02)} = 5.64 \pm e_4$$

$$\frac{1.76\ (\pm 1._7\%) \times 1.89\ (\pm 1._1\%)}{0.59\ (\pm 3._4\%)} = 5.64 \pm e_4$$ $$\%e_4 = \sqrt{(\%e_1)^2 + (\%e_2)^2 + (\%e_3)^2}$$	
$y = x^a \quad \Rightarrow \quad \%e_y = a(\%e_x)$	$y = \sqrt{x} = x^{1/2}\ \left(\frac{1}{2}\right)(2\%) = 1\%$
$y = \log x$	$\frac{e_y}{y} = (\ln 10)\ e_x \approx 2.302\ 6\ e_x$

Table 3-1 Summary of rules for propagation of uncertainty

Function	Uncertainty	Function	Uncertainty
$y = x_1 + x_2$	$e_y = \sqrt{e_{x_1}^2 + e_{x_2}^2}$	$y = x^a$	$\%e_y = a\ \%e_x$
$y = x_1 - x_2$	$e_y = \sqrt{e_{x_1}^2 + e_{x_2}^2}$	$y = \log x$	$e_y = \frac{1}{\ln 10}\frac{e_x}{x} \approx 0.434\ 29\ \frac{e_x}{x}$
$y = x_1 \cdot x_2$	$\%e_y = \sqrt{\%e_{x_1}^2 + \%e_{x_2}^2}$	$y = \ln x$	$e_y = \frac{e_x}{x}$
$y = \frac{x_1}{x_2}$	$\%e_y = \sqrt{\%e_{x_1}^2 + \%e_{x_2}^2}$	$y = 10^x$	$\frac{e_y}{y} = (\ln 10)\ e_x \approx 2.302\ 6\ e_x$
		$y = e^x$	$\frac{e_y}{y} = e_x$

NOTE: x represents a variable and a represents a constant that has no uncertainty. e_x/x is the relative error in x and $\%e_x$ is $100 \times e_x/x$.