U.S. Department of **Health & Human Services**

FDA **U.S. Food and Drug Administration**

Elemental Analysis Manual
for Food and Related Products

The following is a section of the Elemental Analysis Manual for Food and Related Products.

For additional information and to view other sections of the manual, visit the Elemental Analysis Manual for Food and Related Products web page at

https://www.fda.gov/food/laboratory-methods-food/elemental-analysis-manual-eam-food-and-related-products

U.S. Department of **Health & Human Services**

FDA U.S. Food and Drug Administration

Elemental **A**nalysis **M**anual
for Food and Related Products

3.4 Special Calculations

Version 3.1 (December 2021)

Table of Contents

GLOSSARY and ACRONYMS

Section 3.4 provides guidelines for some frequently used, basic calculations. Although harmonization of the terminology, equation variables, and their subscripting is desirable, analysts will find variations because the EAM methods were written at different times by different authors. For example, mass of analytical portion may be expressed as m, m_s, M_x, m_{MTD}, or $M_{analyticalportion}$.

When using an EAM method, it is preferential to follow the terminology, units, and calculational formulas provided in the method, however it is understood that equivalent terminology, units, and calculations formulas do not constitute a "deviation" from the method.

The calculations covered in Section 3.4 include both gravimetric and volumetric approaches with the former receiving preference. It is acknowledged that both are analytically sound but method instructions always take precedence. For example, if a gravimetric approach is specified, calculations shown in Section 3.4 do not allow the use of volumetric procedures instead.

3.4.1. FORTIFICATION RECOVERY

The marginal method is used for fortification recovery. [1]

- Fortified analytical portion (FAP)

$$FAP\ Recovery(\%) = \left[\frac{C_f - C_u}{C_a}\right] \times 100\%$$ 3.4 Equation 1

where: C_f = mass fraction of element measured in FAP (mg/kg)

C_u = mass fraction of element measured in UAP (mg/kg)
(set to zero if <ASDL)

C_a = calculated mass fraction of element added in FAP (mg/kg)

and $$C_a = \frac{C_{fs} \times m_{fs}}{m_{ap} \times MCF}$$ 3.4 Equation 2

where: C_{fs} = mass fraction of element in fortification solution (mg/kg)

m_{fs} = mass of fortification solution added to analytical portion (kg)

m_{ap} = mass of analytical portion (kg)

MCF = mass correction factor (1 if water or other solvent not added to aid homogenization)

Volumetric fortification.

When fortification is added volumetrically, use the following alternate for Equation 2:

$$C_a = \frac{C_{fs} \times V_{fs}}{m_{ap} \times MCF}$$

where: C_{fs} =*concentration of element in fortification solution (mg/L)*

V_{fs} =*volume of fortification solution added to analytical portion (L)*

Conversion between m/v and m/m units are readily accomplished using solution densities.

- Fortified method blank (FMB)

$$FMB\ Recovery(\%) = \left[\frac{C_f - MBK}{C_a}\right] \times 100\%$$
3.4 Equation 3

where: C_f = mass fraction of element measured in FMB (µg/kg)

MBK = mass fraction for Batch MBK (µg/kg; set to 0 if <ASDL)

C_a = calculated mass fraction of element added in FMB (µg/kg)

and
$$C_a = \frac{C_{fs} \times m_{fs}}{m_{MBK}}$$
3.4 Equation 4

where: C_{fs} = mass fraction of element in fortification solution (mg/kg)

m_{fs} = mass of fortification solution added to MBK (kg)

m_{MBK} = mass of MBK (kg)

If fortification solution is in mass/volume units, use the following alternate for Equation 4:

$$C_a = \frac{C_{fs} - V_{fs}}{m_{MBK}}$$

where: C_{fs} = *concentration of element in fortification solution (mg/L)*

V_{fs} = *volume of fortification solution added to MBK (L)*

- Fortified analytical solution (FAS)

$$FAS\ Recovery(\%) = \frac{C_f \times \left(\frac{m_{fs} + m_{AS}}{m_{AS}}\right) - C_u}{C_a} \times 100\%$$
3.4 Equation 5

where: C_f = mass fraction of element measured in FAS (mg/kg)

C_u = mass fraction of element measured in unfortified analytical solution (mg/kg); If (set to zero if <ASDL)

C_a = calculated mass fraction of element added to FAS (mg/kg)

(and)
$$C_a = \frac{C_{fs} \times m_{fs}}{m_{AS}}$$
3.4 Equation 6

where: C_{fs} = mass fraction of element in fortification solution (mg/kg)

m_{fs} = mass of fortification solution added to analytical solution (kg)

m_{AS} = mass of analytical solution (before fortification, kg)

3.4 Equation 5 can be simplified if fortification solution amount is <5% of the amount of the analytical solution being fortified. The dilution effect is small enough for the following simplified equation to be acceptable:

$$FAS\ Recovery(\%) = \left[\frac{C_f - C_u}{C_a}\right] \times 100\% \qquad \text{3.4 Simplified Equation 5}$$

If fortification solution is mass/volume units, use the following alternate for Equation 6:

$$C_a = \frac{C_{fs} \times V_{fs}}{m_{AS}}$$

where: C_{fs} = *concentration of element in fortification solution (mg/L)*

V_{fs} = *volume of fortification solution added to analytical solution (L)*

3.4.2. OTHER RECOVERY

- Reference material (RM)

$$RM\ Recovery(\%) = \frac{C_{meas}}{C_{ref}} \times 100\% \qquad \text{3.4 Equation 7}$$

where: C_{meas} = measured element mass fraction for RM (mg/kg)

C_{ref} = reference element mass fraction (accepted as the "true" value; mg/kg)

- Check solution recovery (or Independent check solution or Continuing calibration verification)

$$Check\ Solution\ Recovery\ (\%) = \frac{C_{meas}}{C_{ref}} \times 100\% \qquad \text{3.4 Equation 8}$$

where: C_{meas} =   measured element mass fraction in check solution (µg/kg)

C_{ref} =   reference element mass fraction in check solution as prepared (µg/kg)

Element level units may be different than given above but must be the same for R and T (e.g., R and T could both be mg/kg or they could both be mg/L).

3.4.3. DILUTION FACTOR

Dilution factor (DF)—factor by which the concentration in a diluted analytical solution is multiplied to obtain the concentration in the analytical solution.

- Gravimetric dilution

$$DF = \frac{m_f}{m_i}$$

3.4 Equation 9

where: m_i =   portion of initial solution (g)

m_f =   final mass (total mass, may be sum of m_i plus mass of diluent; g)

- Volumetric dilution

$$DF = \frac{V_f}{V_i}$$

3.4 Equation 10

where: V_i =   portion of initial solution (L)

V_f =   final volume (total volume, may be sum of V_i plus volume of diluent; L)

For gravimetric dilution/mixing when the element level is in mass/volume and the densities of the initial and final solutions differ significantly (i.e., by more than 5%, or $\rho_i/\rho_f > 1.05$), then use the following equation:

$$DF = \frac{(m_f/\rho_f)}{(m_i/\rho_i)}$$

3.4 Equation 11

where: ρ_i =   Density of initial solution (g/mL)

ρ_f =   Density of final solution (g/mL)

- Serial dilution

Individual gravimetric DFs (or volumetric DFs) can be combined to obtain an overall DF for solutions produced by serial dilution. For example, the DF for an initial solution diluted three times is calculated as follows:

gravimetric   volumetric (volumetric methods only)

$$DF = \left(\frac{m_{f1}}{m_{i1}}\right) \times \left(\frac{m_{f2}}{m_{i2}}\right) \times \left(\frac{m_{f3}}{m_{i3}}\right) \quad DF = \left(\frac{V_{f1}}{V_{i1}}\right) \times \left(\frac{V_{f2}}{V_{i2}}\right) \times \left(\frac{V_{f3}}{V_{i3}}\right)$$

3.4 Equation 12

where: m_{i1} or V_{i1} =   amount of initial solution for first dilution (kg or L)

m_{f1} or V_{f1} =   final amount for first dilution (kg or L)

m_{i2} or V_{i2} =   amount of first diluted solution for second dilution (kg or L)

m_{f2} or V_{f2} =   final amount for second dilution (kg or L)

m_{i3} or V_{i3} =   amount of second diluted solution for third dilution (kg or L)

m_{f3} or V_{f3} =   final amount for third dilution (kg or L)

3.4.4.   CONVERTING UNITS

3.4.4.1. SIMPLE CONVERSIONS

- Conversions with no net change in numerical value

For trivial conversions that result in no net change in the numerical value, the value can be re-written with the new units plus indication that the new expression is either equivalent to (≡) or equal to (=) the former expression.   3.4 Equation 13

> *Example: An instrument gives the result 15.8 μg/g but it needs to be expressed in mg/kg in a report of analysis. An analyst could write the following:*
>
> $$15.8 \; \frac{\mu g}{g} \equiv 15.8 \; \frac{mg}{kg}$$

- Conversions with only movement of decimal point

For simple conversions such as a metric unit change where the only mathematical manipulation is a change in decimal point location, a value can be re-written with the new units plus indication that the new expression is either equivalent to (≡) or equal to (=) the former expression.

> *Example: An instrument gives the result 0.00259 kg but it could be expressed as 2.59 g in an equation.*
>
> $$0.00259 \; kg \equiv 2.59 \; g$$

3.4.4.2. CONVERSIONS ASSOCIATED WITH VOLUMETRIC UNITS

If a standard solution is provided with volumetric units (e.g., mass/volume) then conversion to mass/mass will be necessary for use in methods such as 4.7 that require gravimetric calculations. These conversions require use of density (ρ) which is typically expressed in one of three equivalent formats - g/mL, mg/μL, or kg/L. (Note that 'equivalent' as used here means that the numerical value for ρ is the same regardless of which of these three units are used.)

- Gravimetric preparation of standard solution expressed in volumetric units

To perform gravimetric standard solution preparation when an element level for the initial solution (*e.g.*, stock standard) is expressed in mass/volume units, the density of the initial solution must be known (provided by most commercial manufacturers). If the element level in the final solution must also be expressed in mass/volume units, the density of the final solution must be known as well. For low levels below ~1%, the density can be assumed to be the same as the diluent, which can be determined with sufficient accuracy by measuring the mass of diluent in a tared volumetric flask. The density of solutions can also be determined by using density meter instruments. Both bench top and hand held instruments are available.

Dispense a mass (typically 0.1–1.0 g) of initial solution to nearest 0.0001 g in a tared, clean plastic bottle. Add diluent so that final solution mass (e.g., to 100 g) provides the required element level. The level of each element in the final solution is calculated as follows:

(C_f in mass/mass units) $$C_f = C_i \times \frac{(m_i/\rho_i)}{(m_f)}$$ 3.4 Equation 14

(C_f in mass/volume units) $$C_f = C_i \times \frac{(m_i/\rho_i)}{(m_f/\rho_f)}$$ 3.4 Equation 15

where: C_f = element level in final solution (mg/kg in Equation 14 or mg/L in Equation 15)

C_i = element level in standard solution (mg/L)

ρ_f = density of final solution (g/mL)

ρ_i = density of standard solution (g/mL)

m_f = mass of final solution (g)

m_i = portion of standard solution dispensed (g)

When densities of the initial and final solutions are the same and C_f is desired to be in mass/volume, then the element concentration in the final solution is calculated as follows:

$$C_f = C_i \times \frac{m_i}{m_f}$$ 3.4 Equation 16

- Estimation of mass needed to obtain desired concentration

The following equation provides the mass needed to obtain a desired concentration:

$$m_i = \rho_i \times V_f \times \left(\frac{C_{f*}}{C_i}\right)$$ 3.4 Equation 17

where: C_{f*} = desired concentration of final solution (mg/L)

C_i = concentration of initial solution (mg/L)

ρ_i = density of initial solution (g/mL)

V_f = approximate desired volume of final solution (mL)

m_i = portion of initial solution (g)

For example, if the approximate desired volume is 0.1 L, the desired final concentration is 5 mg/L, and a 1,000 mg/L stock solution has a density of 1.009, then use Equation 17.

$$m_i = 1.009 g/mL \times 100 mL \times \left(\frac{5 mg/L}{1,000 mg/L}\right) = 0.5045 g$$

This mass, within about 10%, is used to prepare the final solution. The analyte concentration in the final solution (S_f) is calculated based on the exact mass of the initial solution taken. Continuing the example above, if the densities of the initial and final solutions are equal and a 0.5548 g portion of initial solution was used and the mass of the final solution was 102.525 g then Equation 16 is used.

$$C_f = 1,000 \text{ mg/L} \times \frac{0.5548 \text{ g}}{102.525 \text{ g}} = 5.4114 \text{ mg/L}$$

- Mass/volume to mass/mass

$$\frac{\frac{mg}{L}}{\rho} = \frac{\frac{mg}{L}}{\frac{kg}{L}} = \frac{mg}{kg} \qquad \text{(or)} \qquad \frac{\frac{\mu g}{mL}}{\rho} = \frac{\frac{\mu g}{mL}}{\frac{g}{mL}} = \frac{\mu g}{g} \qquad \text{3.4 Equation 18}$$

Example: A working standard solution certificate has nickel at 10.01 µg/mL and density 1.045 g/mL. For use in calculations, express Ni in µg/g.

$$\frac{10.01 \frac{\mu g}{mL}}{1.045 \frac{g}{mL}} = 9.579 \ \frac{\mu g}{g}$$

3.4.5.   PERCENT DIFFERENCE

- Relative percent difference (RPD) of two measurements.

Concentration units can be in mass/mass or mass/volume but must be the same for both C_1 and C_2

$$RPD(\%) = \left[\frac{|C_1 - C_2|}{(C_1 + C_2)/2}\right] \times 100\% \qquad \text{3.4 Equation 19}$$

where: C_1 =   concentration for first measurement

C_2 =   concentration for second measurement

- Percent difference (PD) between a measurement result and the reference value

$$PD(\%) = \left[\frac{|C_{ref} - C_{meas}|}{C_{ref}} \right] \times 100\%$$

3.4 Equation 20

where: C_{ref} = reference concentration

C_{meas} = measured concentration

3.4.6. MASS CORRECTION FACTOR (MCF)

Factor applied to analytical portion mass to account for water (or other solvent) added to aid homogenization of analytical sample.

$$MCF = m_{ap}/(m_{ap} + m_w)$$

3.4 Equation 21

where: m_{ap} = mass of analytical portion homogenized (g)

m_w = mass of reagent water (or other solvent) added to aid homogenization (g)

3.4.7. HISTORY

EAM 3.4 Table 1. History

Version	Revisions Made	Effective Date
1.0	*Special Calculations*	June 2008
2.0	Converted to PDF for web posting.	September 2014
3.0	Major update. Renamed to *Converting Units*. Modified many equations and added others to fill gaps associated with differences between gravimetric and volumetric units and mixed measurement (i.e., measurements in one format and calculations in the other).	August 2015
3.1	Updated; added *History* section.	December 2021

References

[1] AOAC International, "Appendix D: Guidelines for Collaborative Study Procedures To Validate Characteristics of a Method of Analysis," in *Official Methods of Analysis of AOAC INTERNATIONAL*, 18th ed., Gaithersburg, MD, USA, 2005.