



U.S. Department of Health & Human Services



U.S. Food and Drug Administration

# Elemental Analysis Manual

## for Food and Related Products

### 3.4 Special Calculations

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#### GLOSSARY

Section 3.4 provides guidelines for some generic calculations to help analysts follow the methods. Attempt has been made for the terminology and equation variables, and their subscripting to be the most commonly used. However, analysts will find variations because the

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EAM methods were written at different times by different authors. For example, mass of analytical portion can be  $m$ ,  $m_s$ ,  $M_x$ ,  $m_{MTD}$ , or  $M_{analytical\ portion}$ . Analysts will therefore need to adjust equation terminology according to the definitions in any given method.

The equations in Section 3.4 do not give flexibility for analysts to deviate from any procedure or calculation given in a method. For example, a volumetric calculation in section 3.4 (i.e., one involving mass/volume units) would be irrelevant for methods such as 4.7 and 4.10 which require gravimetric procedures and calculations.

### 3.4.1 FORTIFICATION RECOVERY

The marginal method of calculating percent recovery is used for fortification recovery calculations.<sup>1</sup>

(1) Fortified analytical portion (FAP)

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

where:  $C_f$  = mass fraction of element measured in FAP (mg/kg)  
 $C_u$  = mass fraction of element measured in UAP (mg/kg)  
 (if  $C_u$  is negative, set it equal to zero for calculation)  
 $C_a$  = calculated mass fraction of element added in FAP (mg/kg)

and  $C_a = (C_{fs} \times m_{fs}) / (m_{ap} \times \text{MCF}) \quad \text{3.4 Equation 2}$

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)

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*For methods that specify a fortification solution in concentration units (NOT for methods with gravimetric procedures): If the element level in the fortification solution is expressed in concentration units (mass/volume) instead of mass fraction (mass/mass), then use the following alternate for Equation 2:*

$$C_a = (C_{fs} \times V_{fs}) / (m_{ap} \times \text{MCF})$$

where:  $C_{fs}$  = concentration of element in fortification solution (mg/L)  
 $V_{fs}$  = volume of fortification solution added to analytical portion (L)

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(2) Fortified method blank (FMB)

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

where:  $C_f$  = mass fraction of element measured in FMB ( $\mu\text{g}/\text{kg}$ )  
 $\text{MBK}$  = mass fraction for Batch MBK ( $\mu\text{g}/\text{kg}$ )  
 $C_a$  = calculated mass fraction of element added in FMB ( $\mu\text{g}/\text{kg}$ )

and 
$$C_a = (C_{fs} \times m_{fs}) / m_{MBK} \quad 3.4 \text{ Equation 4}$$

where:  $C_{fs}$  = mass fraction of element in fortification solution ( $\text{mg}/\text{kg}$ )  
 $m_{fs}$  = mass of fortification solution added to MBK ( $\text{kg}$ )  
 $m_{MBK}$  = mass of MBK ( $\text{kg}$ )

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*For methods that specify a fortification solution in concentration units (NOT for methods with gravimetric procedures): If the element level in the fortification solution is expressed in concentration units (mass/volume) instead of mass fraction (mass/mass), then use the following alternate for Equation 4:*

$$C_a = (C_{fs} \times V_{fs}) / m_{MBK}$$

where:  $C_{fs}$  = concentration of element in fortification solution ( $\text{mg}/\text{L}$ )  
 $V_{fs}$  = volume of fortification solution added to MBK ( $\text{L}$ )

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(3) Fortified analytical solution (FAS)

$$\text{FAS Recovery (\%)} = \left[ \frac{C_f \cdot \left( \frac{m_s + m_{AS}}{m_{AS}} \right) - C_u}{C_a} \right] \times 100 \quad 3.4 \text{ Equation 5}$$

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  $C_u$  is negative, set it equal to zero for calculation.  
 $C_a$  = calculated mass fraction of element added to FAS (mg/kg)

and  $C_a = (C_{fs} \times m_{fs}) / m_{AS}$  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)

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*Simplification option: If the amount of fortification solution used is less than 5% of the amount of the analytical solution being fortified, then the dilution effect is small and the following simplified equations are acceptable:*

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

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

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*For methods that specify a fortification solution in concentration units (NOT for methods with gravimetric procedures): If the element level in the fortification solution is expressed in concentration units (mass/volume) instead of mass fraction (mass/mass), then use the following alternate for Equation 6:*

$$C_a = (C_{fs} \times V_{fs}) / m_{AS}$$

where:  $C_{fs}$  = concentration of element in fortification solution (mg/L)  
 $V_{fs}$  = volume of fortification solution added to analytical solution (L)

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### 3.4.2 OTHER RECOVERY

- (1) Reference material (RM)

$$\text{RM Recovery (\%)} = (R/T) \times 100 \quad 3.4 \text{ Equation 7}$$

where: R = element mass fraction measured for RM (mg/kg)  
 T = true (reference) value for RM (mg/kg)

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

$$\text{Check Solution Recovery (\%)} = (R/T) \times 100 \quad 3.4 \text{ Equation 8}$$

where: R = element mass fraction for element in check solution (µg/kg)  
 T = true mass fraction of element in check solution as prepared (µg/kg)

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*Note: Units for the element level may be different than given above but must be the same for R and T (e.g., could be mg/kg or mg/L).*

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### 3.4.3 DILUTION FACTOR

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

- (1) Gravimetric dilution or mixing.

dilution $DF = \frac{m_f}{m_i}$	mixing $DF = \frac{(m_i + m_d)}{m_i}$	3.4 Equation 9
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where:  $m_i$  = portion of initial solution (g)  
 $m_f$  = final mass (g)  
 $m_d$  = diluent mass (g)

(2) Volumetric dilution or mixing (applicable only for methods with volumetric procedures).

dilution	mixing	
$DF = \frac{V_f}{V_i}$	$DF = \frac{(V_i + V_d)}{V_i}$	3.4 Equation 10

where:  $V_i$  = portion of initial solution (L)  
 $V_f$  = final volume (L)  
 $V_d$  = volume of diluent (L)

*For the unusual situation where a gravimetric dilution/mixing is performed and the element level is given in concentration units (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 11:*

$$DF = \frac{(m_f/\rho_f)}{(m_i/\rho_i)} \quad \text{3.4 Equation 11}$$

where:  $\rho_i$  = density of initial solution (g/mL)  
 $\rho_f$  = density of final solution (g/mL)

(3) Serial dilution

Individual gravimetric DFs (or volumetric DFs for methods using volumetric procedures) 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)	
$DF = \left(\frac{m_{f_1}}{m_{i_1}}\right) \times \left(\frac{m_{f_2}}{m_{i_2}}\right) \times \left(\frac{m_{f_3}}{m_{i_3}}\right)$	$DF = \left(\frac{V_{f_1}}{V_{i_1}}\right) \times \left(\frac{V_{f_2}}{V_{i_2}}\right) \times \left(\frac{V_{f_3}}{V_{i_3}}\right)$	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

Although simple in concept, conversions can be surprisingly confusing.

#### 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 ( $\equiv$ ) or equal to ( $=$ ) the former expression. For example:

$$\frac{\mu\text{g}}{\text{g}} \equiv \frac{\text{mg}}{\text{kg}} \qquad \text{3.4 Equation 13}$$

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

$$15.8 \frac{\mu\text{g}}{\text{g}} = 15.8 \frac{\text{mg}}{\text{kg}}$$

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**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 ( $\equiv$ ) or equal to ( $=$ ) the former expression.

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

$$0.00259 \text{ kg} \equiv 2.59 \text{ g}$$


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#### 3.4.4.2 CONVERSIONS ASSOCIATED WITH CONCENTRATION UNITS

If a standard solution is provided with only concentration units (i.e., 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 ( $\rho$ ) which is typically expressed in one of three equivalent formats -  $\text{g/mL}$ ,  $\text{mg}/\mu\text{L}$ , or  $\text{kg/L}$ . (Note that 'equivalent' as used here means that the numerical value for  $\rho$  is the same regardless of which of these three units are used.)

**Gravimetric preparation of standard solution that is provided with concentration units** - To perform gravimetric standard solution preparation when an element level for the initial solution (e.g., stock standard) is expressed in concentration units (mass/volume), the density of the initial solution must be known and is provided by most commercial manufacturers. If the element level

in the final solution must be expressed also in concentration units, the density of the final solution must be known. It can be assumed the same as the diluent, which can be determined easily 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 (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 (100-270 g) provides the required element level. The level of each element in the final solution is calculated as follows:

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

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

- 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)  
 $\rho_f$  = density of final solution (g/mL)  
 $\rho_i$  = 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:

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

**Estimation of mass needed to obtain desired concentration** - The following equation provides the mass needed to obtain a desired concentration:

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

- where:  $C_{f*}$  = desired concentration of final solution (mg/L)  
 $C_i$  = concentration of initial solution (mg/L)  
 $\rho_i$  = 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.

$$m_i = 1.009 \text{ g/mL} \times 100 \text{ mL} \times \left( \frac{5 \text{ mg/L}}{1,000 \text{ mg/L}} \right) = 0.50450 \text{ g}$$

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 102.5250 g then Equation 16 is used.

$$C_f = 1,000 \text{ mg/L} \times \frac{0.5548 \text{ g}}{102.5250 \text{ g}} = 5.4114 \text{ mg/L}$$


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**Mass/volume to mass/mass** - A value given in concentration units (mass/volume) is converted to mass/mass by dividing by density ( $\rho$ ). The following are two common conversions:

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


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*Example: A working standard solution certificate has nickel at 10.01  $\mu\text{g/mL}$  and density 1.045  $\text{g/mL}$ . For use in calculations, the Ni can be expressed in  $\mu\text{g/g}$ .*

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


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### 3.4.5 PERCENT DIFFERENCE

- (1) Relative percent difference (RPD) of two measurements. Levels can be expressed in mass/mass or mass/volume but must be the same for both  $C_1$  and  $C_2$

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

where:  $C_1$  = element level of first measurement  
 $C_2$  = element level of second measurement

- (2) Percent difference (PD) of a known and calculated value

$$\text{PD (\%)} = \left[ \frac{C_1 - C_2}{C_1} \right] \times 100 \quad 3.4 \text{ Equation 20}$$

where:  $C_1$  = known element level  
 $C_2$  = calculated element level

### 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.

$$\text{MCF} = m_{ap} / (m_{ap} + m_w) \quad 3.4 \text{ Equation 21}$$

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 REFERENCES

1. Official Methods of Analysis of AOAC INTERNATIONAL (2005) 18th Ed., AOAC International, Gaithersburg, MD, USA, Appendix D: Guidelines for Collaborative Study Procedures To Validate Characteristics of a Method of Analysis.