

# Elemental Analysis Manual

## for Food and Related Products

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## 3.4 Special Calculations

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### GLOSSARY

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### 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)

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

where:  $C_f$  = mass fraction of element measured in FAP (mg/kg)  
 $C_u$  = mass fraction of element measured in UAP (mg/kg)  
 $C_a$  = calculated mass fraction of element added in FAP (mg/kg)

and  $C_a = (C_s \times V_s) / (m \times \text{MCF}) \quad 3.4 \text{ Equation 2}$

where:  $C_s$  = concentration of element in fortification solution (mg/L)  
 $V_s$  = volume of fortification solution added to analytical portion (L)  
 $m$  = mass of analytical portion (kg)  
 $\text{MCF}$  = mass correction factor (1 if water or other solvent not added to aid homogenization)

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*Note:  $C_u = 0$  if concentration of element measured in UAP is less than zero.*

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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:  $C_f$  = concentration of element measured in FMB ( $\mu\text{g/L}$ )  
 $\text{MBK}$  = Batch MBK ( $\mu\text{g/L}$ )  
 $C_a$  = calculated concentration of element added in FMB ( $\mu\text{g/L}$ )

(3) Fortified analytical solution (FAS)

$$\text{FAS Recovery (\%)} = \left[ \frac{S_f - S_u}{S_a} \right] \times 100 \quad 3.4 \text{ Equation 4}$$

where:  $S_f$  = concentration of element measured in FAS ( $\mu\text{g/L}$ )  
 $S_u$  = concentration of element measured in unfortified analytical solution ( $\mu\text{g/L}$ )  
 $S_a$  = concentration of element added to FAS ( $\mu\text{g/L}$ )

and 
$$S_a = (C_s \times V_s) / V_{AS} \quad 3.4 \text{ Equation 5}$$

where:  $C_s$  = concentration of element in fortification solution ( $\text{mg/L}$ )  
 $V_s$  = volume of fortification solution added to analytical portion (L)  
 $V_{AS}$  = volume of analytical solution after fortification (L)

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*Note:  $S_u$  equals 0 if concentration of element measured in unfortified analytical solution is less than zero.*

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### 3.4.2 OTHER RECOVERY

(1) Reference material (RM)

$$\text{RM Recovery (\%)} = (R/T) \times 100 \quad 3.4 \text{ Equation 6}$$

where:  $R$  = analytical result for RM ( $\text{mg/kg}$ )  
 $T$  = true (reference) value for RM ( $\text{mg/kg}$ )

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

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

where:  $R$  = analytical result for check solution ( $\mu\text{g/L}$ )  
 $T$  = true concentration of check solution as prepared ( $\mu\text{g/L}$ )

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*Note: Concentration units may be different than given above but must be the same for R and T.*

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### 3.4.3 DILUTION FACTOR

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

- (1) Volumetric dilution Type A (Diluting a volume portion of initial solution to a final volume)

$$DF = \frac{V_f}{V_i} \quad 3.4 \text{ Equation 8}$$

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

- (2) Volumetric dilution Type B (Mixing a volume portion of initial solution with a volume of diluent of the same matrix)

$$DF = \frac{(V_i + V_d)}{V_i} \quad 3.4 \text{ Equation 9}$$

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

- (3) Gravimetric dilution

Gravimetric dilution—practice of quantitatively preparing dilute solutions from more concentrated ones by combining known masses of solution and diluent. A mass of initial solution is mixed with diluent and final mass is measured.

$$DF = \frac{(m_f/d_f)}{(m_i/d_i)} \quad 3.4 \text{ Equation 10}$$

where:  $m_i$  = portion of initial solution (g)  
 $m_f$  = final mass (g)  
 $d_i$  = density of initial solution (g/mL)  
 $d_f$  = density of final solution (g/mL)

When densities of initial solution and diluent are the same then

$$DF = \frac{m_f}{m_i} \quad 3.4 \text{ Equation 11}$$

(4) Serial dilution

Individual volumetric or gravimetric DFs are multiplied to obtain an overall DF for solutions produced by serial dilution. For example, the DF for an initial solution diluted sequentially by three Type A volumetric dilutions is calculated as follows:

$$DF = \left( \frac{V_{f1}}{V_{i1}} \right) \times \left( \frac{V_{f2}}{V_{i2}} \right) \times \left( \frac{V_{f3}}{V_{i3}} \right) \quad 3.4 \text{ Equation 12}$$

where:  $V_{i1}$  = portion of initial solution for first dilution (L)  
 $V_{f1}$  = final volume for first dilution (L)  
 $V_{i2}$  = portion of first diluted solution for second dilution (L)  
 $V_{f2}$  = final volume for second dilution (L)  
 $V_{i3}$  = portion of second diluted solution for third dilution (L)  
 $V_{f3}$  = final volume of third dilution (L)

### 3.4.4 GRAVIMETRIC STANDARD SOLUTION PREPARATION

To perform gravimetric standard solution preparation, the density of the initial solution (*e.g.*, stock standard) must be known and is provided by most commercial manufacturers. The density of the final solution must also be known and can be assumed the same as the diluent, which can be determined easily by measuring the mass of diluent in a tared volumetric flask. 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 concentration. The concentration of each element in the final solution is calculated as follows:

$$S_f = S_i \times \frac{(m_i/d_i)}{(m_f/d_f)} \quad 3.4 \text{ Equation 13}$$

where:  $S_f$  = concentration of final solution (mg/L)  
 $S_i$  = concentration of initial solution (mg/L)  
 $d_f$  = density of final solution (g/mL)  
 $d_i$  = density of initial solution (g/mL)  
 $m_f$  = mass of final solution (g)  
 $m_i$  = portion of initial solution dispensed (g)

When densities of the initial and final solutions are the same, then

$$S_f = S_i \times \frac{m_i}{m_f} \quad 3.4 \text{ Equation 14}$$

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*Estimation of mass needed to obtain desired concentration:*

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

$$m_i = d_i \times V_f \times \left( \frac{S_f}{S_i} \right) \quad 3.4 \text{ Equation 15}$$

where:  $S_f$  = desired concentration of final solution (mg/L)  
 $S_i$  = concentration of initial solution (mg/L)  
 $d_i$  = 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 and the desired final concentration is 5 mg/L, then for a 1,000 mg/L stock solution with a density of 1.009 then*

$$m_i = 1.009 \text{ g/mL} \times 100 \text{ mL} \times \left( \frac{5 \text{ mg/L}}{1,000 \text{ mg/L}} \right) = 0.50450 \text{ g} \quad 3.4 \text{ Equation 16}$$

*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*

$$S_f = 1,000 \text{ mg/L} \times \frac{0.5548 \text{ g}}{102.5250 \text{ g}} = 5.4114 \text{ mg/L} \quad 3.4 \text{ Equation 17}$$


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### 3.4.5 PERCENT DIFFERENCE

- (1) Relative percent difference (RPD) of two measurements

$$\text{RPD (\%)} = \left[ \frac{|C_1 - C_2|}{(C_1 + C_2)/2} \right] \times 100 \quad 3.4 \text{ Equation 18}$$

where:  $C_1$  = concentration of first measurement  
 $C_2$  = concentration of second measurement

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

$$\text{PD (\%)} = \left[ \frac{C_1 - C_2}{C_1} \right] \times 100 \quad 3.4 \text{ Equation 19}$$

where:  $C_1$  = known concentration  
 $C_2$  = calculated 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.

$$\text{MCF} = m_s / (m_s + m_w) \quad 3.4 \text{ Equation 20}$$

where:  $m_s$  = mass of analytical sample homogenized (g)  
 $m_w$  = mass of reagent water (or other solvent) added to aid homogenization (g)

## 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.