

# Elemental Analysis Manual

## for Food and Related Products

### Archive Notes

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## 4.0.2 Method Performance Checks

Version 1.0 (June 2008)  
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### GLOSSARY

#### 4.0.2.1 REFERENCE MATERIAL (RM)

RM results are used to assess accuracy. Assessing accuracy using a z-score (§3.5.3) is the preferred procedure. However, for simplicity, control limits are usually used and set at an RM recovery of  $100 \pm 20\%$  unless the RM's reference uncertainties (at 95% confidence level) are greater than 20%. For each element of interest, there must be an established value for that element at a concentration above LOQ. If three or more RMs are analyzed then only two-thirds of an element's RM recovery results must meet the control limit. Repeat analysis of all batch analytical solutions if control limit is exceeded. If RM recovery fails again, reject batch results and repeat digestion and analysis of all samples in batch. When appropriate RMs are unavailable other quality control measures are used to judge acceptance of batch analytical results (e.g., FAP recovery, FMB recovery).

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*Note: Failing the control limit should be highly unusual because a laboratory's experience analyzing a RM should establish predictable results. Whenever a new RM is investigated, treat it initially as an unknown and if accuracy is a problem, identify and correct the cause(s) of the problem(s) before the RM is used as a control material for judgment of batch quality. In-house RMs with established values are acceptable.*

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*Note: Choice of RM depends on availability but should be similar to the sample matrix. Unfortunately, suitable RMs may not be obtainable. Non-certified element concentrations provided on a certificate may be used for quality control if the laboratory has established the ability to meet the acceptance criteria.*

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#### 4.0.2.2 FORTIFIED ANALYTICAL PORTION (FAP)

FAP results are used to assess analyte recovery and matrix induced interference. Control limits are usually a FAP recovery of  $100 \pm 20\%$ . A poor recovery can indicate analyte loss during preparation, physical/transport interference and spectral interference. Prepare two replicate portions, one portion is the unfortified analytical portion (UAP) and the other portion, the FAP, is fortified with each analyte before digestion. Fortify by pipetting no more than a total of 1 mL (0.5 mL for CV-AAS) of fortification solution(s) (may be measured gravimetrically) into the digestion vessel. Fortification level is based on prior knowledge of analyte concentration in the sample or typical reported levels for the food. Fortification should be 100-200% of expected analyte level. Foods with low analyte levels or unknown analyte levels should be fortified so that analyte concentration in the analytical solution is approximately midpoint of LDR or for ICP-AES between 10 times ASQL and 40 mg/L. Analyze an FAP for each food type for which there is no FAP recovery data on record using the method. If FAP recovery results are unacceptable, re-analyze the FAP analytical solution. If FAP recovery fails again then the analyst must use other batch analytical results to evaluate the quality of the analysis and determine if the batch samples must be re-analyzed. Unreliable FAP recovery results may occur due to measurement imprecision when the fortification level is less than 100% of the native level. When fortification levels are too low, the FAP recovery may be considered invalid due to an inappropriate fortification level. In this case, other quality control measures may be used to judge acceptance of batch analytical results (e.g., RMs, FMBs). Depending on the other quality control results and the purpose of the analysis (i.e., survey or enforcement) another FAP may need to be analyzed using an appropriate fortification level based on the sample's analyte concentration.

#### 4.0.2.3 FORTIFIED ANALYTICAL SOLUTION (FAS)

A FAS is used to assess matrix-induced interference. Control limits are usually a FAS recovery of  $100 \pm 10\%$ . If FAS is out of control, suspect matrix interference, dilute analytical solution by a factor of 2 or more and re-analyze. FAS fortification, recovery check, dilution and re-analysis can usually be performed automatically by the instrument's software and autosampler. As an alternative to dilution, if FAS recovery was  $\geq 50\%$ , analytical solution can be analyzed by method of standard additions. If recovery was  $< 50\%$  then there is a possibility that the standard additions technique may not be able to compensate for the large matrix interference present.

#### 4.0.2.4 FORTIFIED METHOD BLANK (FMB)

FMB checks accuracy of the fortification procedure without any matrix effects. Control limits are usually a FMB recovery of  $100 \pm 10\%$ . If FMB is out of control, an error in fortification should be suspected and FMB needs to be prepared again and re-analyzed. The FMB is an optional quality control sample but can be helpful in verifying the fortification procedure and reveal pipet malfunctions and dilution errors.

#### 4.0.2.5 LABORATORY MBK ( $MBK_L$ )

Mean of analyte concentration measurements (to at least 3 significant digits) of at least 5 independently prepared MBKs (unfortified) rounded to a two significant-digit number.  $MBK_L$  should be established using MBK results accumulated from many independent analyses over extended periods (*i.e.*, months).  $MBK_L$  is determined for each analyte-method-instrument combination.  $MBK_L$  represents the analyte level expected during routine analyses and MBKs analyzed with a batch of samples are compared to  $MBK_L$ .

- $MBK_L$  is subtracted from all analytical solutions results.

#### 4.0.2.6 LABORATORY MBK CRITICAL VALUE ( $MBK_C$ )

Mean of analyte concentration measurements (to at least 3 significant digits) of the MBKs (unfortified) used to establish  $MBK_L$  plus 2 times the standard deviation of these MBKs rounded up to the next greatest two significant-digit number.  $MBK_C$  is used to judge the quality of MBKs analyzed with each batch of samples.

$$MBK_C = MBK_L + (2 \times s)$$

where,  $s$  is the standard deviation of the MBKs used to establish  $MBK_L$ .

#### 4.0.2.7 BATCH METHOD BLANKS (MBK)

MBK results are used to assess contamination from the laboratory environment and reagents. A batch's MBK results are compared to the expected level of  $MBK_L$ . A batch's MBK results are acceptable when at least two-thirds of the MBK results are  $\leq MBK_C$ . Batch MBKs exceeding this  $MBK_C$  should be uncommon. The more frequently batch MBKs exceed  $MBK_C$ , the more attention should be directed to identifying and correcting the cause of contamination or to consider reestablishing  $MBK_L$  and  $MBK_C$ .